STUDY OF THE CHARACTERISTICS OF THE CORROSION FILM ON ZIRCONIUM USING POLARIZED LIGHT

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Cambridge, Massachusetts

May 16, 1952

Secretary of the Faculty
Massachusetts Institute of Technology
Cambridge, Massachusetts

Dear Sir:

In accordance with the requirements for the Degree of Master of Science, we submit herewith a thesis entitled, "Study of the Characteristics of the Corrosion Film on Zirconium using Polarized Light."

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STUDY OF THE CHARACTERISTICS OF THE CORROSION FILM ON ZIRCONIUM USING POLARIZED LIGHT

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1952

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ABSTRACT
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STUDY OF THE CHARACTERISTICS OF THE CORROSION FILM ON ZIRCONIUM USING POLARIZED LIGHT

by

Commander Nels Roland Nelson, U. S. Navy

and

Lieutenant John Wade Heintz, U. S. Navy

Submitted for the degree of Master of Science in the Department of Physics, Massachusetts Institute of Technology on May 16, 1952

While pure zirconium is virtually non-corrosive at low temperatures, its corrosion-resistance at high temperatures varies over a wide range depending upon the amount and type of impurities present. The purpose of this thesis was to study with polarized light the effect of nitrogen on the corrosion-resistance of zirconium in distilled water at 460°F and at 560°F.

Six specimens of zirconium (essentially the same in chemical analysis except for variations in nitrogen content) were studied with plane polarized light. The data obtained consisted of measurements of the phase shift and rotation of the plane of polarization of the light reflected from the surfaces of the specimens during a series of incremental corrosion film growths.

The characteristic angle of zirconium in the electropolished condition was observed to be 293° -08' ± 1° -39'. In general, with successive

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additions of corrosion film, the characteristic angle decreased for each specimen. An exception is made to this statement for those specimens which had their optic axes normal to the surface.

Previous work in this field has shown that an increase in nitrogen causes a decrease in the corrosion-resistance of zirconium. The results of the studies made in this thesis do not indicate that either the characteristic angle, Ψ, (or any other observable parameter) vary in a manner consistent with nitrogen content. Consequently, without additional investigation, polarized light cannot be used to test the corrosion-resistance of a random zirconium sample.

With refined methods of reducing the amount of miscellaneous impurities in the zirconium specimens to be studied, with reduction of errors in the optical equipment, and with the incorporation of a preferred crystal orientation, it is possible that polarized light may still be used to determine the corrosion-resistance of zirconium.

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Chapter I

INTRODUCTION

Because zirconium has a low cross-section for thermal neutrons and has desirable structural qualities, it is being used more and more in nuclear reactors. In this application, the zirconium often is exposed to relatively high temperature distilled water for long periods of time. Consequently, its corrosion-resistance under those conditions has become important, especially since it varies over a wide range depending upon the amount and type of impurities present. To date, no reliable test has been developed which will give a rapid, accurate evaluation of the corrosion-resisting quality of a random sample of zirconium.

The purpose of this thesis was to study with polarized light the effect of nitrogen on the corrosion-resistance of Westinghouse Grade I Crystal Bar Zirconium. The particular type of corrosion investigated was that caused by surface contact with distilled water at 450° F and at 560° F. The study was limited to areas of single grains which did not include any grain boundaries.

Modern research metallographs provide an excellent means of using polarized light to examine the grain structure of hexagonal metals such as zirconium. The optical effects involved in such an examination of the reflection of polarized light from opaque surfaces have been known for over a half-century. More recently, the development of an Elliptical Vibration Compensator has made possible an easy method of accurately

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measuring these effects. A detailed discussion of the theory involved in this method is contained in Chapter II.

While a survey of the literature has revealed no previous attempts to use polarized light to study zirconium corrosion films, a small amount of information is available concerning related problems. Most of this information is summarized by B. W. Mott and H. M. Haines[7] in "The Examination of Metals Under Polarized Light," Part I, AERE, M/R 604. The latest previous work of this nature is a Bachelor of Science thesis entitled "High Sensitivity Measurements of the Optical Anisotropy of Beryllium" by Edward L. Bronson at M.I.T. in 1951. He found appreciable changes in elliptical compensator readings for different types of films on beryllium. This result suggested to Dr. A. R. Kaufmann that the characteristics of the corrosion film of zirconium might be studied in a similar manner and that possibly the results might provide a means for devising a corrosion-resistance test. Preliminary work by the authors during the M.I.T. fall semester supported this belief and precipitated the present study.

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Chapter II

THEORETICAL ANALYSIS

At room temperature zirconium is a close packed hexagonal crystal. The optical properties of such crystals are known to vary for different directions in the crystal and are therefore said to be optically anisotropic. In the hexagonal system there are two crystallographic planes of symmetry -- one containing the major crystallographic axis or optic axis, c; and the plane that is normal to this containing the basal plane. These two directions are called the principal directions of the crystal. The optical properties of the crystal in each of these directions can be described by its refractive index, n, and absorption coefficient, k. Thus n_o and k_o describe the optical characteristics of the crystal in the direction of the optic axis and n_b and k_b describe the characteristics in a direction perpendicular to the optic axis.

When a ray of plane polarized light falls upon the polished surface of a zirconium specimen, the vibrating vector of this light can be resolved into two components -- one lying in the plane containing the optic axis and the other perpendicular to this. Since all experimental work carried out in this thesis was done with normal vertical illumination on zirconium specimens, all figures and descriptions will be based on this. Figure II-1 shows the ray of plane polarized light at normal incidence to a zirconium specimen whose optic axis is neither in the same plane as the normal to the surface nor in the plane containing the vibrating electric vector of the incident light. E_i is the incident electric vector with component E_{io} along the optic axis and E_{ib} perpendicular to the optic axis.

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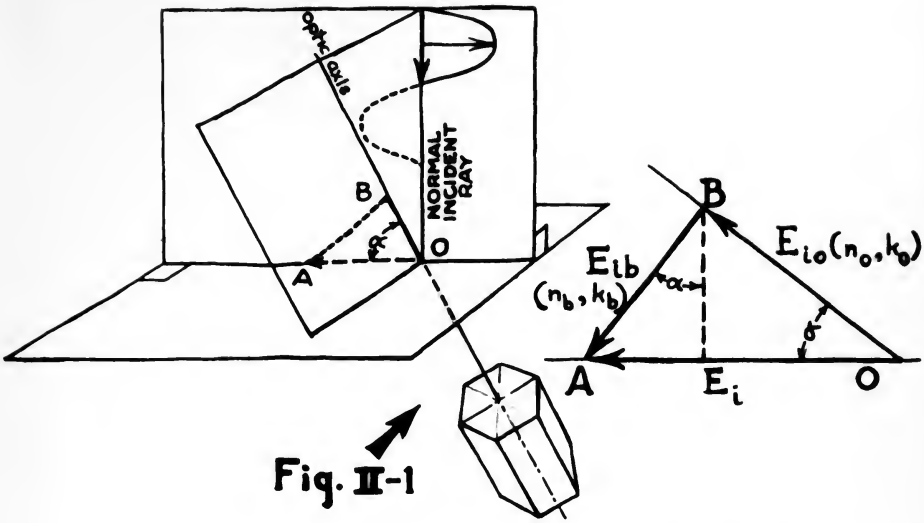

Fig. II-1

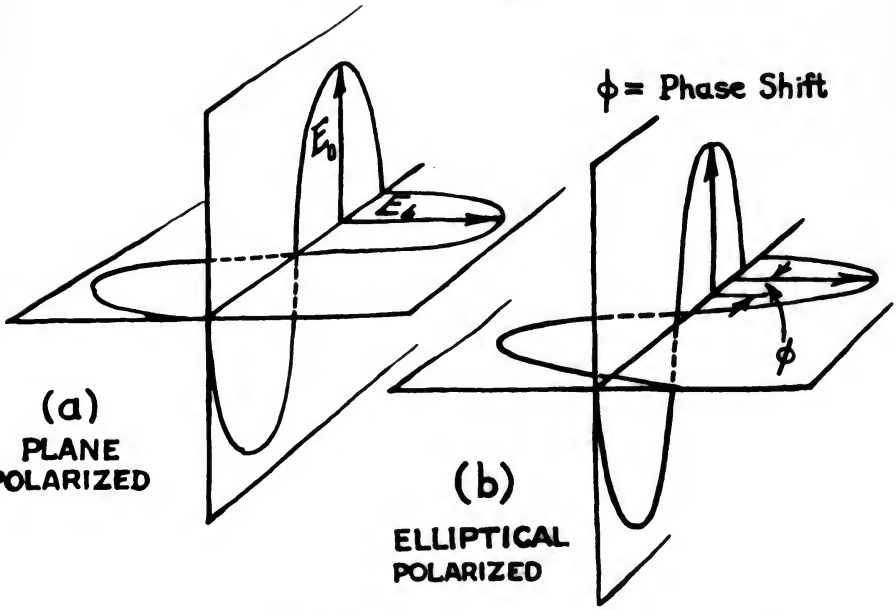

Fig. II-2

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The refractive index, n, and the absorption coefficient, k, are properties of the crystal which affect the intensity or energy carried by the incident wave. Since the energy of a plane polarized light wave is proportional to the square of the electric vector, the electric vector, E_i, and its components will be correspondingly affected. The properties, n and k, in a specific direction are referred to as an index pair -- being n_b, k_b and n_o, k_o for their respective directions in the crystal. These index pairs may be thought of as vector operators. In zirconium which is an opaque (absorbing) crystal, these index pairs or vector operators are complex operators of the form (n-jk).

Since the index pair, n_o, k_o is different than n_b, k_b, the incident component E_{ib} will be operated on differently than the component E_{io}. If the reflecting power of a principal direction is defined as the ratio of the intensity of the reflected component to the intensity of the incident component, we have

$$R_o = \frac{I_{ro}}{I_{io}} = \frac{(E_{ro})^2}{(E_{io})^2} \quad \text{and}$$

$$R_b = \frac{I_{rb}}{I_{ib}} = \frac{(E_{rb})^2}{(E_{ib})^2} \qquad \text{----- (1)}$$

or in terms of the index pairs n_o, k_o and n_b, k_b when the medium of immersion is air, equation (1) becomes[4]

$$R_o = \frac{(n_o - 1)^2 + n_o^2 k_o^2}{(n_o + 1)^2 + n_o^2 k_o^2}$$

$$R_b = \frac{(n_b - 1)^2 + n_b^2 k_b^2}{(n_o + 1)^2 + n_b^2 k_b^2} \qquad \text{----- (2)}$$

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Thus when the incident vector component, E_{io}, is operated on by R_o during reflection, the reflected component, E_{ro}, will be different. Similarly when E_{ib} is operated on by R_b, E_{rb} will be different. Both components of the incident vector will have been changed in magnitude and will be out of phase with each other when reflected. That is, while in the specimen the change in velocity of propagation of E_{io} due to R_o was not the same as the change introduced in E_{ib} by R_b and upon emergence the reflected components were out of phase with each other.

In Figure II-2(a) the incident ray is shown resolved into two components at right angles to each other and in Figure II-2(b) the reflected ray is shown resolved into two components at right angles to each other. Thus the incident plane polarized light is reflected as elliptically polarized light due to the optical anisotropy of zirconium.

In the special case where the electric vector, E_i, is coincident with one of the principal directions such as the optic axis, there will be no component in the direction of the basal plane, i.e., E_{ib} will be zero. E_{io} will equal E_i and the resulting reflected light will be linearly polarized. Summarizing, when plane polarized light is incident (normal) on a specimen of zirconium, elliptically polarized light will be reflected except when the direction of polarization is coincident with a principal direction. This can be better visualized in Figure II-3. In Figure II-3(a), component E_o is operated on by R_o and E_b by R_b. This changes E_o and E_b so that they form elliptically polarized light. For each angle α, the resulting elliptical light will be different and the maximum ellipticity will occur for $\alpha = 45°$. In Figure II-3(b) component E_o is operated on by R_o and since $E_b = 0$, the reflected light will be linearly polarized in the same direction.

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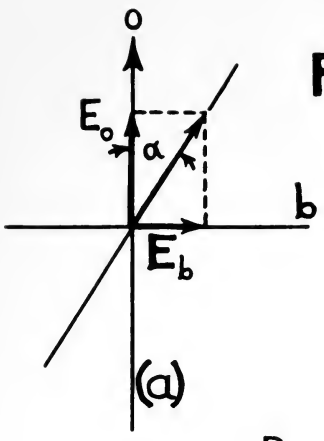
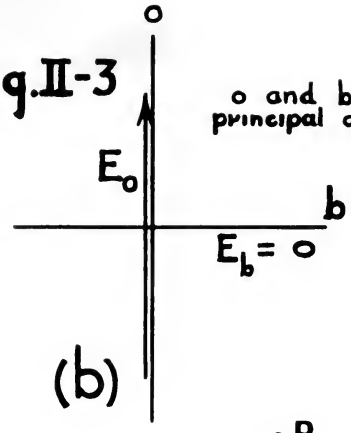


Fig.II-3



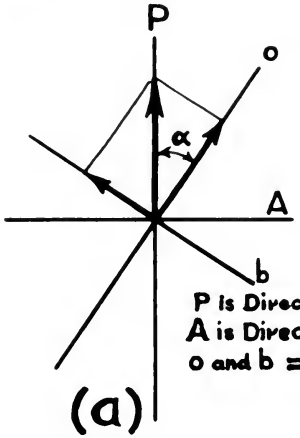
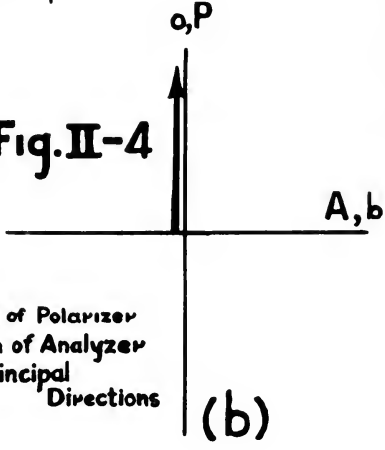


Fig.II-4



P is Direction of Polarizer
A is Direction of Analyzer
o and b = Principal Directions

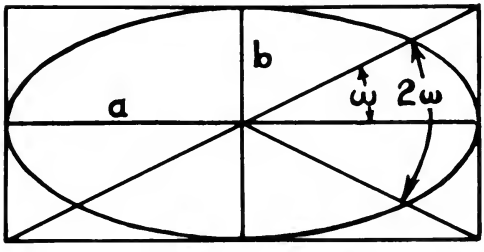


Fig.II-5

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Now consider the case in which the specimen is viewed through an analyzer whose direction is at right angles to the polarizer supplying the light. This is a condition known as "crossed nicols." Figure II-4(a) shows the direction of the polarizer, P, the direction of the analyzer, A, and the principal directions of the crystal, c and b. When the specimen is rotated to an angle α between P and c, the light reflected to the analyzer will be elliptically polarized and the components of this light parallel to the direction of the analyzer will pass through. As α varies when the stage is rotated the ellipticity of the reflected light and thus the components parallel to the analyzer direction will change. This will vary the intensity of the reflected light as viewed through the analyzer. When the direction of polarization is coincident with the principal direction, c, as in Figure II-4(b) the reflected light is plane polarized and is at right angles to the direction of the analyzer. No light should be transmitted in this case. Therefore as the stage with the specimen is rotated through 360° there should be four points of minimum intensity and four points of maximum intensity — the minimum occurring when the principal directions are coincident with the polarizer direction and the maximum when the ellipticity is the greatest, i.e., when the angle α is 45° (diagonal position).

It is noted, however, during observations that the minimum intensities are never complete nulls as would be expected and that the four maximum intensities are not of equal magnitude. As brought out by Berek[4], with physical optical systems it is almost impossible to provide a source of pure plane polarized light to the specimen. The optical systems

usually create a small amount of ellipticity and therefore even when the principal directions are coincident with the direction of polarization as in Figure II-4(b), some light can be viewed through the analyzer.

If devices, which can compensate for the ellipticity introduced by zirconium into plane polarized light, can be inserted in the path of the elliptical light, it is believed that much information can be obtained about the surface properties of zirconium. This ellipticity is caused by a phase shift and the rotation of the plane of polarization. Therefore the ellipticity could be removed by two compensating devices of known characteristics -- one which removes the phase shift and the other which removes the rotation. To see how this can be done, the geometry of elliptically polarized light should be reviewed.**

The locus of a point tracing an elliptical path is given by

$$\left.\begin{aligned} y &= R \sin \frac{2\pi t}{T} \\ x &= A \sin \left(\frac{2\pi t}{T} + \phi \right) \end{aligned}\right\} \qquad \text{---------(3)}$$

where the x component leads the y component by an angle ϕ (in radians). R is the amplitude of the y component (retarded) and A is the amplitude of the x component (accelerated). T is the period of vibration and t is the time elapsed since $y = 0$.

Referring to Figure II-5, the ellipticity is defined as

$$\delta = \frac{b}{a} = \tan \omega \qquad \text{---------(4)}$$

or

$$\sin 2\omega = \frac{2\delta}{1 + \delta^2} \qquad \text{---------(5)}$$

** The figures and nomenclature used are those of C. A. Skinner[3].

and the equation of an ellipse referred to its own axes is

$$\frac{x^2}{a^2} + \frac{y^2}{b^2} = 1 \qquad \text{- - - - - - - - (6)}$$

In Figure II-6, consider an elliptical pattern of light where x is the incident vibration direction. Θ is defined as the azimuth of the elliptical light.

$$\frac{x'^2}{a^2} + \frac{y'^2}{b^2} = 1 \qquad \text{- - - - - - - - (7)}$$

and $$A = x_1' \cos \Theta - y_1' \sin \Theta \qquad \text{- - - - - - - - (8)}$$

Combining (7) and (8)

$$A^2 = a^2 \cos^2 \Theta + b^2 \sin^2 \Theta \qquad \text{- - - - - - (9)}$$

$$R^2 = a^2 \sin^2 \Theta + b^2 \cos^2 \Theta \qquad \text{- - - - - - (10)}$$

adding and subtracting (9) and (10)

$$A^2 + R^2 = a^2 \quad b^2 \qquad \text{- - - - - - (11)}$$

$$A^2 - R^2 = (a^2 - b^2)(\cos^2 \Theta - \sin^2 \Theta)$$

$$= (a^2 - b^2) \cos 2\Theta \qquad \text{- - - (12)}$$

By introducing the phase lead, φ, of the A component over the R component, other fundamental relations can be derived. From equation (3), if $y = 0$ and $x = p$, we have

$$p = A \sin \varphi \qquad \text{- - - - - - - - (13)}$$

From Figure II-6, this point p is x_2', y_2' and

$$x_2' = p \cos \Theta = A \sin \varphi \cos \Theta \qquad (14)$$

$$y_2' = p \sin \Theta = A \sin \varphi \sin \Theta \qquad (15)$$

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Substituting (14) and (15) into (6) gives

$$A^2 \sin^2 \varphi = \frac{a^2 b^2}{a^2 \sin^2 \theta + b^2 \cos^2 \theta} \qquad --(16)$$

and from (10) $$AR \sin \varphi = ab \qquad ---------(17)$$

We have further

$$\cos^2 \theta = 1 - \sin^2 \theta = 1 - \frac{a^2 b^2}{(a^2\cos^2\theta + b^2\sin^2\theta)(a^2\sin^2\theta + b^2\cos^2\theta)}$$

or $$\cos^2 \varphi = \frac{(a^2 - b^2)^2 \sin^2 \theta \cos^2 \theta}{A^2 R^2}$$

or $$AR \cos \varphi = \frac{a^2 - b^2}{2} \sin 2\theta \qquad --------(18)$$

Rewriting equations (11), (12), (17), and (18) and defining

$$P \equiv \frac{A^2 + R^2}{2} = \frac{a^2 + b^2}{2} \qquad ------(19)$$

$$Q \equiv \frac{A^2 - R^2}{2} = \frac{a^2 - b^2}{2} \cos 2\theta \qquad ------(20)$$

$$K \equiv AR \cos \varphi = \frac{a^2 - b^2}{2} \sin 2\theta \qquad ------(21)$$

$$S \equiv AR \sin \varphi = ab \qquad ------(22)$$

Combining (19), (20), (21), (22) gives

$$Q^2 + K^2 + S^2 = P^2 \qquad ------(23)$$

which suggests the equation for a sphere in which Q, K, and S are the rectangular coordinates whose radius is P with the center at the origin.

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To show how the sphere may be used to determine the constants of an elliptic vibration we have for the azimuth θ of the ellipse from (20) and (21)

$$\tan 2\theta = \frac{K}{Q} \qquad \text{- - - - - - - - - - - (24)}$$

in which θ can assume any value between 0 and π and is independent of S. Similarly from (19) and (22)

$$\frac{2ab}{a^2 + b^2} = \frac{S}{P} \qquad \text{- - - - - - - - - - - (25)}$$

and since $\delta = \frac{b}{a}$ from (4)

$$\frac{2\delta}{1 + \delta^2} = \frac{S}{P} \qquad \text{- - - - - - - - - - - (26)}$$

and from (5) $$\frac{S}{P} = \sin 2\omega \qquad \text{- - - - - - - - - - - (27)}$$

so $$\delta = \tan \omega \qquad \text{- - - - - - - - - - - (28)}$$

Therefore the ellipticity, δ, is independent of Q and K and since $\sin 2\omega$ can have any value between -1 and +1, δ may range between -1 and +1.

Further, the phase lead ϕ of any A component, from (21) and (22) is given by

$$\tan \phi = \frac{S}{K} \qquad \text{- - - - - - - - - - - (29)}$$

In Figure II-7, let the QK plane be the equatorial plane of the sphere and the S axis be the polar axis. Let longitude be measured from the Q axis eastward, and the latitude upward from the equatorial plane. Inspection shows that 2θ is the longitude and 2ω is the latitude of the

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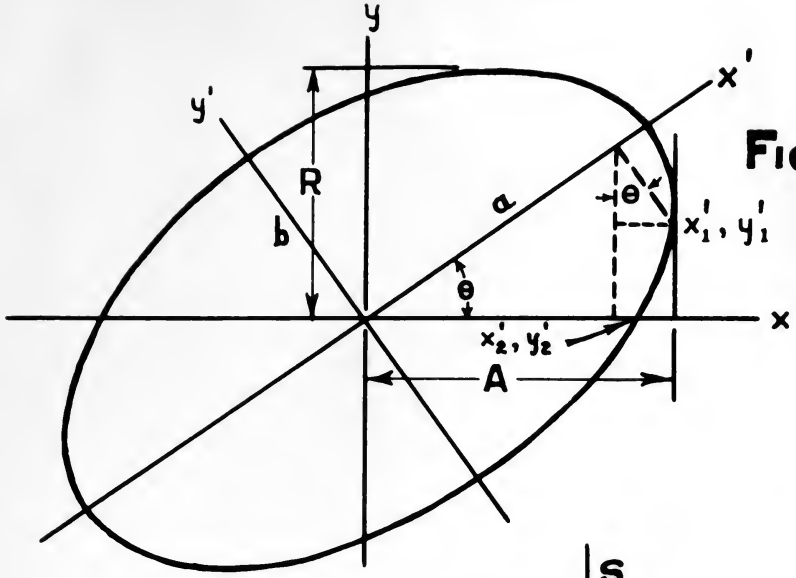

Fig. II-6

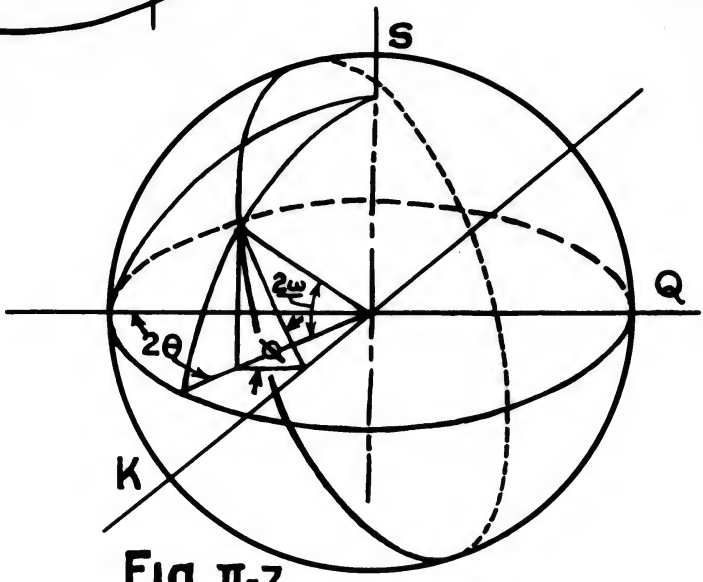

Fig. II-7

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expressed vibration. Therefore the longitude on this sphere of reference can be used to determine the azimuth of the vibration while its latitude can be used to determine ellipticity or phase shift. Plane polarized light (no ellipticity) is represented by the sum total of points on the equator while positive and negative circular polarized light are represented by the north and south poles respectively.

The Bausch and Lomb Elliptical Vibration Compensator was used in this thesis to measure the rotation and ellipticity of the reflected light. In the compensator unit there are two elements: a rotation compensator and an elliptical compensator. Figure II-8 shows a schematic diagram of the compensator unit.

The rotation compensator will be taken up first. The rotation compensator consists of two identical glass prisms cemented together with a transparent cement of the same index as that of the glass. On the inclined inner face of one prism is a quarter-wave interference with an index differing from that of the glass. This quarter wave interference is tin oxide applied by high vacuum thermal evaporation. This quarter-wave is for a specific wave length and a correction must be made if other wave lengths of light are to be used. Referring to Figure II-8, it is seen that the inclined film of the rotation compensator transmits the p-component of the incident polarized light more copiously than the s-component. This results in a rotation of the polarized light traversing the film.

$$\tan \psi_d = \frac{D_p}{D_s} \quad \text{and} \quad \tan \psi_e = \frac{E_p}{E_s}$$

$$\tan \psi_d = R \tan \psi_e \quad \text{where} \quad R = \frac{D_p E_s}{D_s E_p}$$

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If the azimuth of the incident light on the film is ψ_e and the azimuth of the transmitted light is ψ_d, then with no mica plate present, ψ_d would also be the azimuth of the light incident on the specimen. If the specimen produces a rotation of the incident light, the reflected light will have an azimuth $\psi_{e'}$ and after passing through the compensator film the light will be rotated again and emerge with an azimuth of $\psi_{d'}$. Again

$$\tan \psi_{d'} = R \tan \psi_{e'}$$

With the polarizer and analyzer crossed, compensation is obtained by turning the film about the optical axis of the system until extinction occurs. At this point compensation has been effected. Therefore at compensation with the compensator setting at r,

$$r = \psi_e = \psi_{d'}$$

The rotation produced by the specimen is $(\psi_d - \psi_{e'})$.

$$\tan (\psi_d - \psi_{e'}) = \frac{\tan \psi_d - \tan \psi_{e'}}{1 + \tan \psi_d \tan \psi_{e'}}$$

$$\tan (\psi_d - \psi_{e'}) = \frac{R^2 - 1}{2R} \sin 2r$$

or
$$\epsilon = \frac{R^2 - 1}{2R} \sin 2r \qquad \text{- - - - - - - (30)}$$

where
$$\tan (\psi_d - \psi_{e'}) = \epsilon$$

Since the rotation produced by the specimen is usually much less than 5°

$$\epsilon \approx (\psi_d - \psi_{e'}) = \frac{R^2 - 1}{2R} \sin 2r \qquad \text{- - - - (31)}$$

In Figure II-8 the elliptical compensator is located above the rotation compensator and consists of an extremely thin mica plate cemented between cover glasses and is rotatable in its own plane. As in the rotation compensator, light traverses the mica plate in both the incident and reflected directions, thereby removing half the phase shift due to the specimen in the incident beam and the other half from the reflected beam. Since the relationship of elliptically polarized light to a sphere has already been shown, it is easy to present a geometrical explanation of what happens to light as it traverses the elliptical compensator.

Figure II-9 shows the five operations on the incident plane polarized light. The fast axis (principal direction) of the specimen is assumed to be at 45° to the direction of polarization. This will be a longitude of 90° in the sphere since all angles are double. (It should be recalled that the maximum ellipticity due to the specimen occurs when the direction of polarization of the incident light is at 45° to one of the principal directions of the crystal. This will fix the position of the specimen on the stage in the procedure to be described later).

Figure II-9(a) shows the rotation of the incident light after passing through the rotation compensator (inclined quarter-wave film). Figure II-9(b) shows the phase shift introduced into the rotated plane polarized light after having passed through the mica plate of the elliptical compensator. This is the light which falls on the specimen. It is to be noted that this light is now slightly elliptical having been given both a rotation and a phase shift. Figure II-9(c) shows the rotation and phase shift given to the incident light by the specimen. Figure II-9(d) shows the phase shift given to the light as it passes back through the

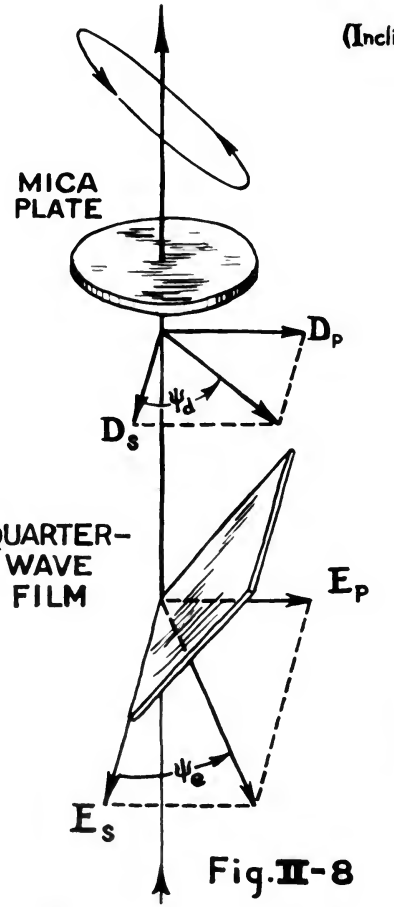

Fig. II-8

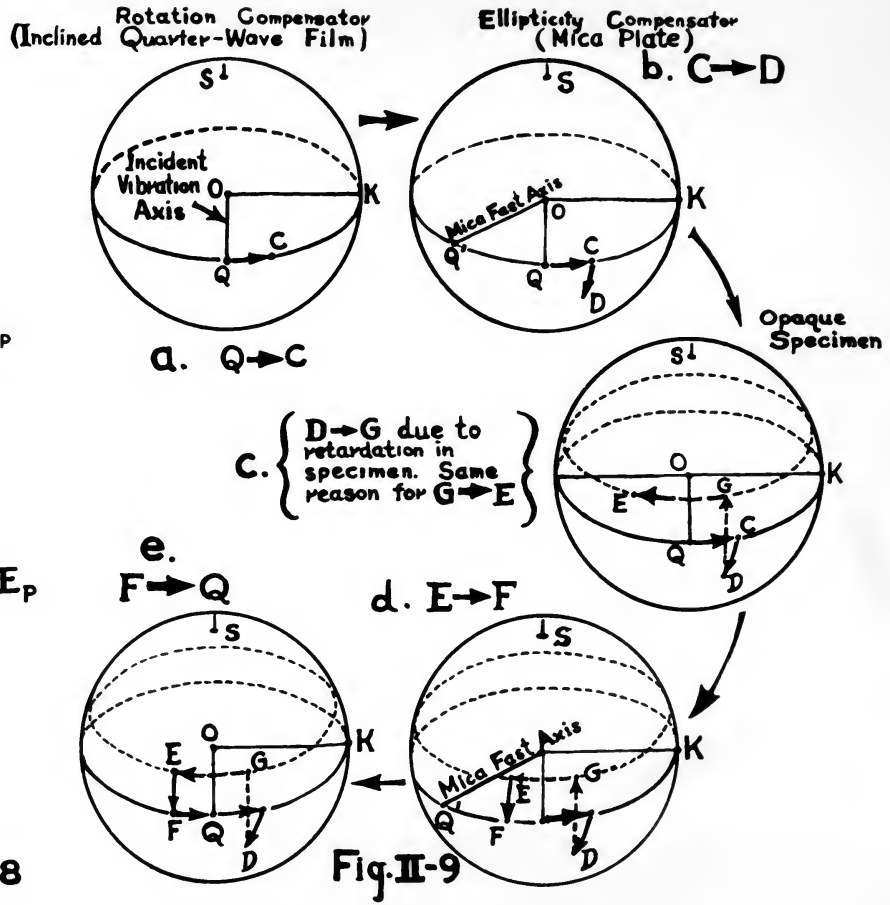

Fig. II-9

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mica plate and Figure II-9(e) shows the rotation added when it passes through the quarter wave film again. This last point lies on the equator of the sphere indicating that it is plane polarized light (no latitude) and is in the same direction as the incident light (no change in longitude). Thus the ellipticity has been compensated for and when viewed through a crossed analyzer there should be extinction.

From Figure II-9, it can be shown by projection geometry (see Appendix A) that

$$\sin \Delta \cong \sin \varphi \left[\sin(2m + 2d_1) + \sin(2m - 2d_2) \right] \quad - - - (32)$$

where Δ is the phase shift on the sphere due to the specimen

m is the angular displacement of the mica plate fast axis from zero

φ is the phase shift due to the mica plate

d_1 and d_2 are individual rotations of incident and reflected beams due to rotation compensator.

Knowing that d_1 and d_2 are small and about equal and that Δ is a small angle, we obtain

$$\Delta \cong 2 \sin \varphi \sin 2m \quad - - - - - - - - - (33)$$

where Δ is in radians.

When the specimen is in the diagonal position, the incident light is reflected with maximum ellipticity. This elliptical light has components vibrating parallel and perpendicular to the direction of polarization. These two components will be out of phase since the light is elliptical. The phase angle between these components which is caused by the specimen is called τ, the characteristic angle.

For each type of specimen there are many anisotropy parameters. These consist of special functions of the index of refraction, the coefficient of absorption, the ellipticity of the waves penetrating into the

specimen, and the index of the immersion medium. However, all of these functions are related to two of the parameters. One will be called the polarization coefficient ϵ and the other the phase coefficient δ. It has been shown that ϵ is the tangent of the azimuth of the major axis of the reflected vibration ellipse measured from the vibration direction of the incident light (Eq. 50) and δ is the ratio of the minor axis to the major axis. For incident linearly polarized light, the reflected ellipse can be written in the form

$$\nu e^{i\tau} = \epsilon + i\delta \qquad \text{----------(34)}$$

where ν is the relative anisotropy and is equal to $\sqrt{\epsilon^2+\delta^2}$

$$\tan \tau = \frac{\delta}{\epsilon} \qquad \text{----------(35)}$$

on the sphere; $$\delta = \tan \omega \qquad \text{----------(28)}$$

and ω is one-half the latitude of a point on the sphere.

Since we are referring to the phase shift (change of latitude) due to the specimen (Figure II-9(c)).

$$-\delta = \tan \frac{\Delta}{2} \qquad \text{(minus due to convention on sphere)}$$

or for small angles of Δ

$$\Delta = -2\delta \qquad \text{----------(36)}$$

From Figure II-8 and Equation (50) it is seen that the tangent of angle between the major axis of the ellipse and the incident plane light in terms of the compensator is

$$\tan (\psi_a - \psi_{a'}) = \epsilon = \frac{R^2-1}{2R} \sin 2r \qquad (50)$$

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Substituting equations (56) and (50) into equation (55), gives

$$\tan \tau = \frac{-\sin \phi \sin 2m}{\frac{R^2 - 1}{2R} \sin 2r} \qquad ---- (57)$$

However, the quarter-wave film in the rotation compensator is exact only for one wave length of light. For wave lengths longer or shorter than this, the light will suffer a relative phase change in passing through. Therefore equation (57) must be corrected for the wave length of light used and becomes

$$\tan \tau = \frac{-\sin \phi \sin 2m + \sin \rho \sin 2r}{\frac{R^2 - 1}{2R} \sin 2r} \qquad (58)$$

where the quantities ϕ, ρ, and R are determined experimentally and are constants of the compensator.

It should be noted in the above derivation that the τ used is not strictly the characteristic angle since the incident light is not plane but elliptical due to passing through the rotation and elliptical compensators once. However, for practical purposes, the approximation is not too bad.

The Bausch and Lomb Elliptical Vibration Compensator (Figure III-8) has calibrated divided circles upon which can be read the settings on the rotation and elliptical compensators. The readings for extinction on two successive diagonal positions of the specimen are subtracted to give the values of 2m and 2r to be used in equation (58).

τ is a unique value — it is a constant for a particular anisotropic metal or mineral regardless of the angle of the basal plane or cleavage plane (provided that the surface condition is the same in each case). The value of τ for isotropic crystals is not constant and varies

between -90° to $+90^{\circ}$. For anisotropic crystals τ will show a change with surface characteristics which are undetectable under ordinary microscopic examination. The measurement of τ serves as a criterion of the excellence of the polishing technique - the highest value of τ being the best polished. Tabulated values showing this fact are available.[9]

If an increase in the characteristic angle, τ, can be used as a criterion for the excellence of polishing techniques, then decreasing angles of τ could serve to indicate a film growth on a polished surface. Therefore, the authors propose to study and attempt to determine the corrosion characteristics of zirconium based on theory described in this section.

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Chapter III

PROCEDURE AND EQUIPMENT

A. Preparation of Specimens

The original material from which all specimens were prepared was an 18" piece of Westinghouse Grade I Crystal Bar Zirconium, approximately 5/8" in diameter. The existing surface film was removed by wirebrushing and by chemical etching in a 1:1 solution of water and nitric acid plus a few drops of concentrated hydrofluoric acid. When all of the film had been removed, the bar was washed under tap water.

From this original bar, a four-inch billet (approximately equal to 100 grams) was cut from the center section. This billet was inspected again for any surface film, especially in and around the jagged grain boundaries of the surface crystals and then it was rewashed and dried in acetone.

In order to homogenize this "mother billet," it was drip melted twice under a vacuum of 10^{-5} microns in a quartz tube using high frequency induction heating as shown in Fig. III-1. The quartz tube was evacuated by the usual laboratory fore pump, diffusion pump vacuum system. The zirconium billet was dripped into a standard M.I.T. copper crucible which is shown in cross section in Figure III-2. Previous work had shown that copper pickup from such a crucible was negligible. After each of the first few drip-meltings, the quartz tube (Figure III-1) showed a slight dark film deposit on the inside surface in the vicinity of the position where the zirconium had been suspended during the melt. This film is believed to be caused by condensation of the miscellaneous minor

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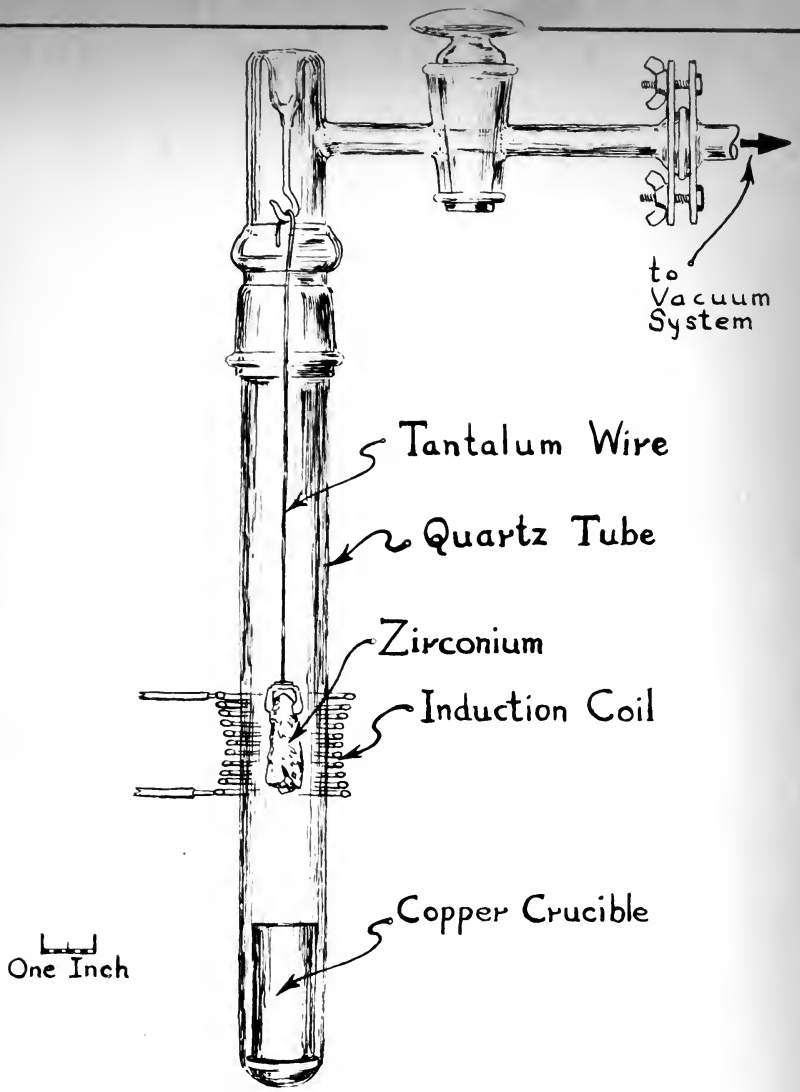

VACUUM DRIP MELTING APPARATUS

Fig. III – 1

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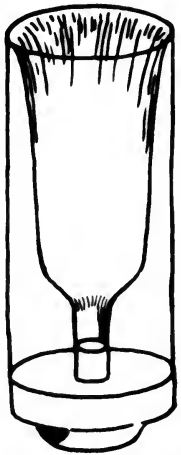

Fig. III-2

Copper Crucible
FULL SIZE

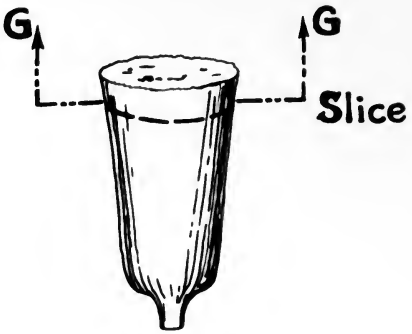

Fig. III-3

Zirconium Billet after having been Drip Melted into Copper Crucible

Fig. III-4

Sectioning of Slice G-G to obtain specimen H

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impurities (e.g., Fe) volatilized during the melting process. It was noted that the amount of film deposited after each melt was less than for the previous melt. This suggested that the major percentage of the impurities of the "mother billet" were volatilized in the first few drip-melts.

Since, from previous experience, a vacuum of 10^{-5} microns was set as the amount of evacuation necessary to avoid appreciable oxygen pickup during any high temperature process involving zirconium, the word "vacuum" throughout the rest of this paper will mean a vacuum of 10^{-5} microns.

After the "mother billet" had been drip melted twice, it was held under vacuum at 1600° C for about ten minutes in order to further homogenize the material. The first specimen (piece H, Fig. III-4) then was roughed out from a 1/8" thick slice cut off from the large diameter end of the drip melted billet as shown in Figures III-3 and III-4.

Piece (H) of Figure III-4 was prepared for metallographic examination while the remainder of the slice was given to the chemist for analysis. Upon completion of the chemical analysis, the material then remaining was subjected to a corrosion-resisting quality test in distilled water at 315° C for approximately 800 hours. Samples A, C, D, and F were so tested.

After the first slice had been cut from the main billet, nitrogen was added to the remainder of the billet. From this billet the second slice was cut yielding the second sample. This process was repeated until six samples had been prepared with increasing quantities of nitrogen. After each nitrogen addition, the billet was drip-melted in vacuum twice to provide homogeneity. A sample calculation to determine the pressure of nitrogen to be bled into the vacuum system in order to add a desired number of parts per million of nitrogen to the billet is given in Appendix B. The six samples so prepared with their nitrogen content in parts per

million are chemically described in Table C-1 of Appendix C and were assigned the following symbols:

Specimen	ppm Nitrogen
A	6
B	70
C	115
D	147
E	150
F	240

The six specimens listed were prepared for metallographic examination as follows[11]:

(a) Paper polished using kerosene on successively finer papers from 320 A down to 3/0 making each pass at 90° to the preceding pass.

(b) Slightly etched in a solution of equal parts of water and nitric acid plus a few drops of hydrofluoric acid, washed in water and dried in acetone.

(c) Electropolished in a solution of one part of 60% perchloric acid to ten parts of glacial acetic acid using a stainless steel anode and approximately 0.3 amperes and 18 volts for about 45 seconds. Experience and skill is needed in this phase in order to produce a "standard" electropolished surface, free from any macroscopic film and surface irregularities. Since a chemical etch film can be produced if the sample is left in the solution after the current is turned off, the sample always was withdrawn from the solution with the current still on. Moreover, since electropolishing for an

excessively long period of time or with excessively high voltage can cause surface deviations, these conditions had to be avoided. Two of the samples inadvertently were excessively electropolished on the first try and had to be paper polished, etched and electropolished over again. All six samples when completed, however, showed a remarkably "standard" surface. The values of the characteristic angle for all of the anisotropic zirconium crystals selected did not differ by more than ±2. Moreover, after corroding these specimens fifteen times, the surfaces again were prepared in the same manner and gave reproducible results of the characteristic angle of the same crystals, within the same limit of error. After each electropolishing operation, the surface was washed with denatured ethyl alcohol and immediately hot-air dried.

B. Metallographic Procedure and Equipment

A detailed description of the equipment used is included in S.B. thesis, M.I.T., 1951, by E. L. Bronson.[10] A photograph of this equipment is shown in Figure III-7.

The general procedure followed was to use an Elliptical Vibration Compensator to measure the phase shift and rotation in the reflected polarized light from a particular portion of a selected crystal after successive growths of corrosion film. In order to do this, it was necessary to examine each specimen very carefully under polarized light in order to select one or more crystals in that specimen which had the following characteristics:

(a) Large enough (1/32" x 1/32" minimum).

(b) Unique irregularities in shape of boundaries to permit easy identification.

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(c) Free from spots or areas which became alternately bright and dark but not in phase sequence with the background crystal material.

(d) Free from twin bands.

(e) As far as possible, free from pits, inclusions, and spots which remained dark throughout an entire rotation.

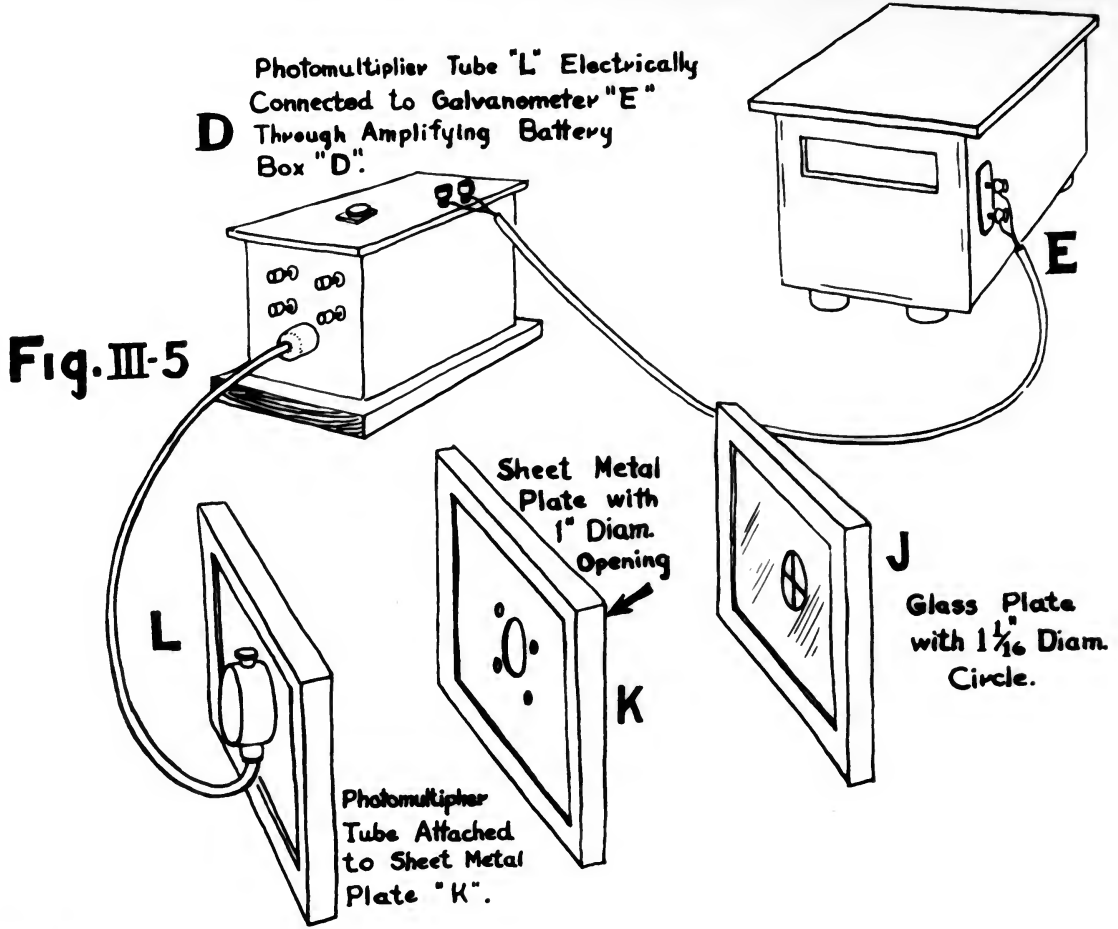
(f) As far as possible, free from shaded variations in color that appear within a crystal on some angles of rotation while appearing uniform in color on other angles. This "mottled effect" previously has shown up in drip-melted samples, but its nature and cause still remain unknown.

Two crystals were selected in each of samples A and F, while one crystal was selected in each of samples B, C, D, and E. The two crystals in A and F were chosen in order to determine whether the orientation of the basal planes had any effect on the variation in the phase shift and in the rotation of the plane of polarization from the surface of the randomly oriented crystals and from the corrosion films grown thereon.

Once the eight crystals were selected, they were photographed at 50 X in order to preserve a record of their contours to assist in identification after the corrosion films had been grown.

Having chosen a particular crystal in each sample, it was necessary to select a particular area in each sample and to devise a means which would permit observations of that particular area (and only that area) after each successive autoclaving. The equipment shown in Fig. III-5, pieces J, K, and L were designed to accomplish this function.

In Figure III-5, piece J is a frosted glass plate set in a metallograph photographic frame. A 1-1/16" diameter circle and perpendicular



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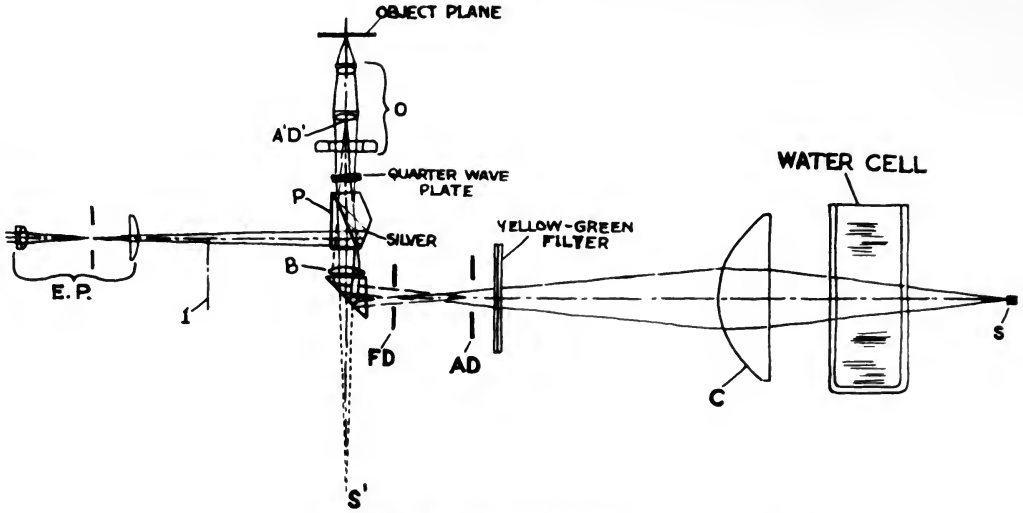
"cross hairs" were drawn in black ink at the center of the glass plate. Next a sheet metal plate with a one-inch circular hole cut out of its center was fitted into another frame so that by superposition, the hole was concentric with the 1-1/16" circle drawn on the glass plate. Four 5/32" bolting holes were drilled as shown in K of Figure III-5. To this plate the housing for the photomultiplier tube[12] was bolted as shown in L of Figure III-5. The metallograph was now ready to be used to measure the intensity of polarized light as reflected from the specimens. Figure III-6 is a diagram of the optical system in the Bausch and Lomb Research Metallograph, which was used in this study. The Carbon Arc source of light normally supplied with the metallograph previously had been replaced with a 40-watt Zirconarc tube which, after a warm-up period of three hours, provided a source of white light which was reasonably constant in intensity. To assist the Zirconarc in maintaining constant intensity, a Raytheon 100 power factor 120 volt 120 watts voltage stabilizer was connected into the alternating current feed to the Zirconarc Power Supply Box. This Power Supply Box acted as a converter and furnished the Zirconarc tube with approximately 2 amperes at 30 volts, direct current. All of the equipment described in this paragraph is shown in Figure III-7.

To make certain that the polarized light always struck perpendicular to the surface of the specimen (or its film), each specimen was viewed with its electropolished side down on a thin glass plate which had been made to fit the recess in the head of the metallograph. This glass plate had a 1/4" hole cut at the center with the edges of the hole carefully ground over so that no protruding sharp edges were available to scratch the surface to be studied.



Bright-field Illumination

S – Source
C – Condenser Lens
AD – Aperture Diaphragm
FD – Field Diaphragm

S' – Virtual Image of Source
B – Condenser Lens
P – Polarizing Vertical Illumination Prism

A'D' – Image of AD
O – Objective and Glass Base
1 – Position of Swing-Out Prism
EP – Camera Eyepiece.

Fig. III-6

METALLOGRAPH OPTICS

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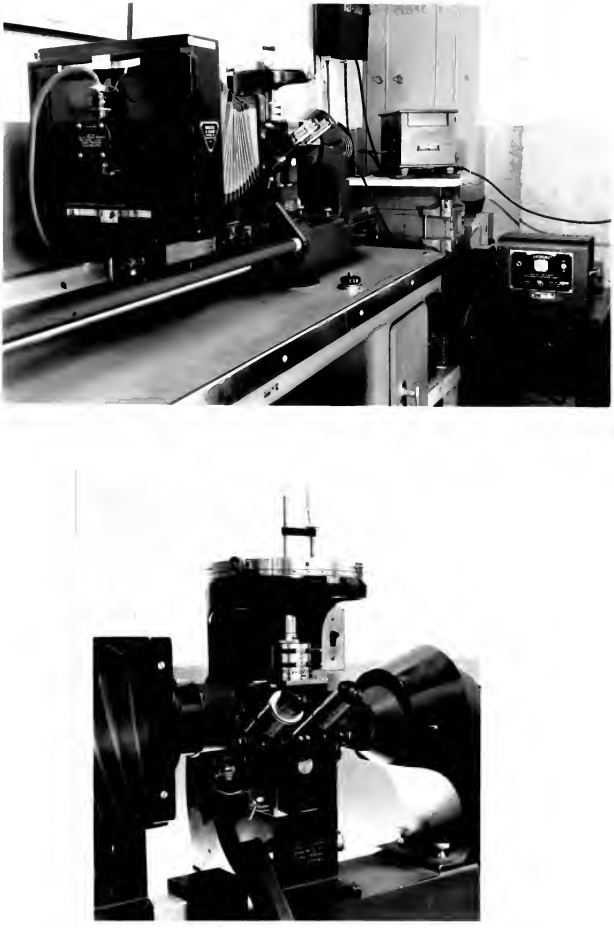

FIGURE III-7

METALLOGRAPH AND ASSOCIATED EQUIPMENT WITH ELLIPTICAL COMPENSATOR IN PLACE

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FIGURE III-8

ELLIPTICAL VIBRATION COMPENSATOR

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The specimen was then centered over the hole in the glass plate so that the metallograph beam fell on the desired crystal. This was most expeditiously accomplished by scanning the sample with a relatively low magnification (5.6 X) objective lens in the metallograph. Once the crystal was located, a 13.5 X objective lens was inserted in place of the 5.6 lens. With a combination of a 13.5 objective lens and a 10 X hyperplane eyepiece, and the bellows extended to 38.6 cm, the magnification obtained was 200 X. Once the cross hairs of the glass plate (J), Figure III-5, were located in a "clean" area of the crystal, a map of the exact location of the cross hairs and the 1-1/16" circle relative to the adjacent grain boundaries was traced on a transparent paper. With this map, it was relatively easy to reposition the crystal after each autoclaving so that the photomultiplier tube was measuring light intensity from the same area each time. It also was necessary to align the objective lens in the metallograph for each crystal so that upon rotation of the stage through 360° the crystal would revolve concentrically around the centering cross hairs. This alignment was most easily and most accurately accomplished by viewing the rotation at 200 X on the glass plate.

Once the crystal had been located accurately, the metallograph stage was rotated in increments of 10° through 360°. The amplification of the photomultiplier circuit[12] was adjusted at the battery box to give readings on the galvanometer ranging between 0 and 100. Instead of tabulating the readings, they were more rapidly plotted directly on reproducible graph paper forms printed especially for this purpose (Appendix D). This portion of the data was taken in order to locate the four azimuthal positions of maximum intensities of the reflected light during a full

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rotation. Henceforth these four positions will be called the diagonal positions of the crystal. The entire curve was recorded, instead of just the peaks, since this plot permitted a graphical location of the exact diagonal positions more accurately than could be obtained merely by sweeping with the galvanometer in the vicinity of the peaks. As it turned out, other interesting physical phenomena became apparent after studying and comparing the shape of the intensity curves.

Having determined the four angles of setting of the metallograph stage for the diagonal positions, the 13.5 objective lens was removed and the Elliptical Vibration Compensator was inserted in its place. The compensator was centered in the same manner as was the 13.5 lens and the exact area to be scanned again was located on the glass plate (J) Figure III-5. This was necessary because each time a lens was removed from the metallograph and then replaced, it was found that its optical axis would not return exactly to its previous position. Having centered the elliptical compensator and relocated the area on the sample with the aid of the tracing, the metallograph table was set on each of the azimuths of the diagonal positions at which points the elliptical compensator was adjusted for extinction. Figure III-8 is a photograph of the elliptical compensator and Figure II-8 is a sketch of its optical system. In Figure III-8 the upper pin controls phase shift while the lower pin controls rotation of the plane of polarization. To adjust the compensator for extinction both pins are rotated by trial and error until the galvanometer gives a minimum reading. The significance of this operation is described in Chapter II. Two extinction readings were taken at each of the four diagonal positions. The phase shift and rotation due to the specimen are included on each graphical form sheet in Appendix D.

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C. Autoclaving Procedure

Since the basic problem consisted of using polarized light to study the characteristics of the corrosion film on zirconium, it was important to be able to correlate the data obtained with some corrosion parameter. Previous work by the authors had shown that 100° C was too low and 315° C was too high for optimum growth of corrosion film for this type of study. Consequently, during run #1, successive autoclavings were carried out at a temperature of 235° C for equal increments of 15 minutes each. In run #2 the time of each autoclaving remained 15 minutes but the temperature was raised to 295° C.

The autoclaving for run #1 is described in detail as follows. The stainless steel autoclave was filled with distilled water and the six specimens were placed in the water with electropolished surfaces facing upwards. The cover was then bolted and the autoclave was heated in an electric oven from room temperature to 235° C using a constant setting on a Variac in the circuit. The time required to bring the autoclave up to temperature in this manner was approximately two hours and twenty-two minutes, and this time remained reasonably constant varying by not more than ±8 minutes. As soon as the autoclave temperature reached 235° C, the autoclave immediately was transferred to a preheated oven with automatic controls which maintained the autoclave temperature at 235° C. After 15 minutes at this temperature, the autoclave was removed, sprayed with water until this sprayed water no longer turned to steam and then immersed in tap water. The entire cooling procedure took approximately five minutes and was constant to within ± 1/2 minute. Once cooled, the autoclave was opened, the samples removed and air-dried with a syringe. For each successive autoclaving, new distilled water was used. Calibrated Chromel-Alumel thermocouples correct to ± 1° C were used.

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Run #2 was conducted in a manner similar to run #1 except that only one autoclaving was done and its temperature was 295° C. The time required to bring the autoclave up to 295° C was 2 hours and 58 minutes. The cooling time was 5 minutes.

In run #1, the corrosion film varied in color from a pale yellow after the first autoclaving, on through straw, to medium brown to bluish for the last autoclaving. In general the changes in film color were similar to those obtained in tempering steel. Polarized light readings were obtained up through the bluish films.

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Chapter IV

RESULTS AND CONCLUSIONS

1. The values obtained for either 2m, 2r, or τ do not vary in any manner consistent with the nitrogen content in the specimens. Therefore, at present this method does not appear suitable for use as a corrosion-resistance test for zirconium.

2. With further refinement in methods of sample preparation and with more accurate optical systems, it still may be possible to use polarized light to determine corrosion-resistance of zirconium. The details of what refinements are necessary and what optical errors should be eliminated are included in Chapter V.

3. Three possible explanations of why the value of τ did not vary as the nitrogen content in the specimens are as follows:

   A. As shown in Appendix C, the inadvertent impurities in the specimens were considerable compared to nitrogen content. For example, in the low nitrogen sample 1/-1 there was about twenty times as much iron and carbon as there was nitrogen. In the remainder of the samples, as the content of nitrogen was increased, volatilization reduced the quantities of other impurities. It is entirely possible that the variation of the effects on corrosion of these other impurities was greater than that of the nitrogen.

   B. Some metals corrode at different rates along different directions. Zirconium, being anisotropic in other properties, probably is also anisotropic in corrosion. Therefore, with random orientation of the eight crystals studied, the effect of variable corrosion

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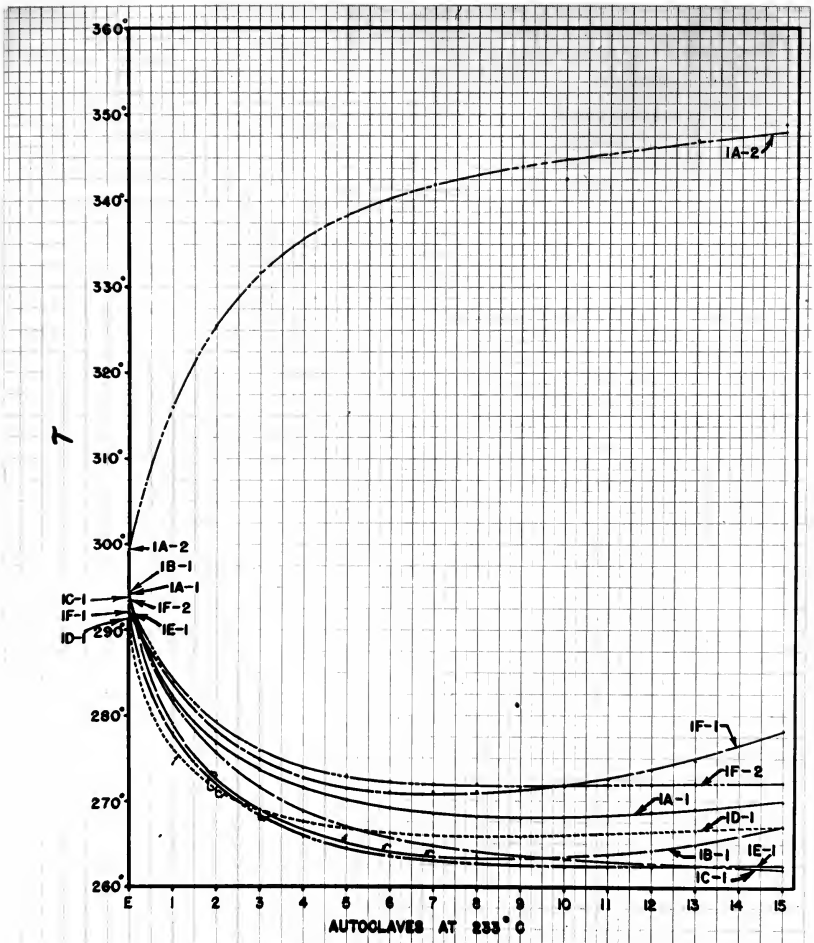

SUMMARY PLOT OF CHARACTERISTIC ANGLES (τ) FOR RUN NO. 1

Fig IV-1

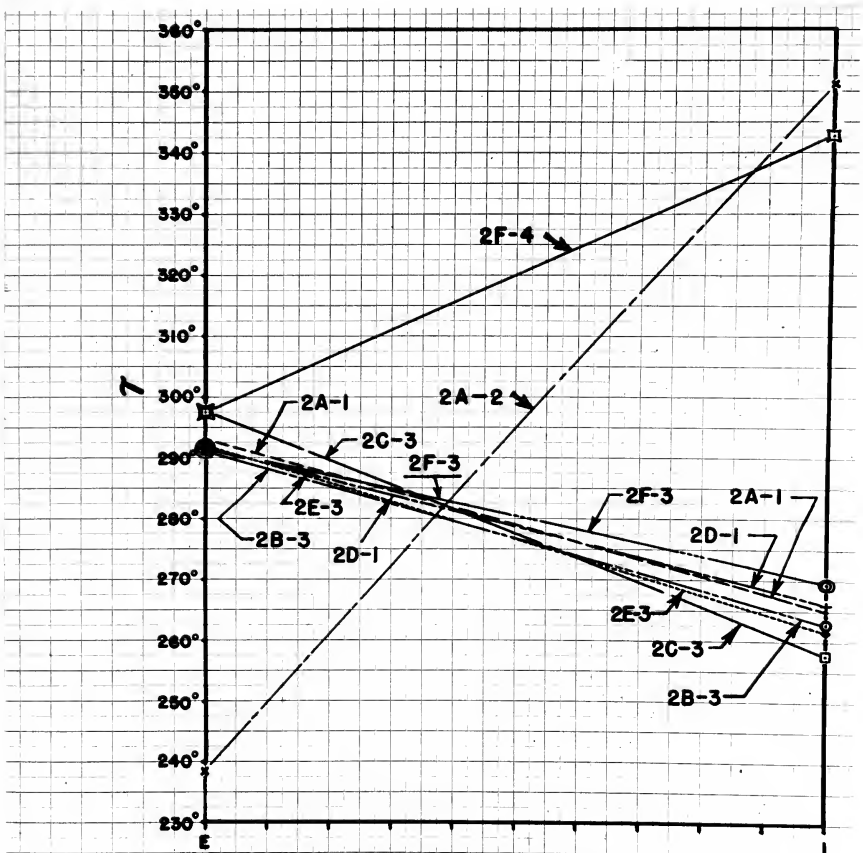

Fig IV-2

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growth caused by grain orientation may have been of greater magnitude than the effect of nitrogen content.

C. A combination of (A) and (B) acting together.

4. The anomalous behavior of curves 1A-2, 2A-2 and 2F-4 are explained by the fact that their optical axes were nearly perpendicular to the surface of the specimen. This statement is based on the fact that the specimens, when rotated upon the stage did not go through sharp maxima and minima of intensity, but instead remained fairly dark throughout the entire rotation. Since a hexagonal crystal has two of its three crystal axes of equal magnitude and since these two equal axes were contained in the surface of the specimen, there was very little ellipticity introduced into the incident plane polarized light. Consequently, the reflected polarized light contained practically no ellipticity which resulted in practically no phase shift. The rotation of the plane of polarization, however, was considerably more than for the other specimens. Hence with essentially a constant phase shift and an increasing rotation of the plane of polarization, the characteristic angle Ψ, which depends on the ratio of the sines of these two parameters, increased rapidly as shown in Figures IV-1 and IV-2.

5. As the corrosion time was increased, the color of the specimens progressed from natural to straw, to yellow, to brown, to blue in overall appearance.

6. Looking at each specimen in more detail, the color of the film was not homogeneous across the surface of any specimen. For example, in a specimen where the overall appearance of the surface gave a brownish appearance, upon more detailed observation some areas

appeared yellow and some blue, whereas the majority appeared brown. The boundaries between these changes in color were sharp and had a general appearance similar to the boundaries of large regular zirconium crystals.

7. Additional zirconium crystal bar samples (not the A to F series discussed in this thesis) were corroded for eighty hours at 315° C plus six days at 345° C. A small amount of white powderish film became noticeable. This film was concentrated in white lines which appeared to be outlining zirconium crystal boundaries.

8. After the 15th autoclaving at 233° F (end of Run #1) the specimens were autoclaved at 233° C for about 40 hours. Upon metallographic examination the film was studded with reddish irregular spots which shone through the much more darkly colored background of the crystal. In addition, there were sets of longitudinal lines which appeared as cracks in the film. In this condition, the diagonal positions of maximum intensities could not accurately be determined, nor could the adjustments be made for minimum intensities on the elliptical compensator. Since the surface being studied was no longer homogeneous, these results were taken as being random and were discarded. From this experience it is felt that polarized light cannot be used to obtain quantitative effects on film growth after the film has either broken down in local areas, or split, or grown too thick.

9. The crystal boundaries remained the same shape and size when observed throughout the entire growth range of film growth. This fact indicates that the film was thin or that its structure was similar to the parent crystal structure.

10. Upon calibration of Elliptical Vibration Compensator #37 with a zirconarc source of white light, the new constants obtained are contained in the following corrected formula for the characteristic angle, $\mathcal{T}_p$

$$\tan \mathcal{T} = -.6666 \frac{\sin 2m}{\sin 2r} + .0208$$

11. When specimens 1A-2, 2A-2 and 2F-4, (which had their optical axes almost normal to the surface), were rotated through 360°, only two points of maximum intensity appeared. Upon investigation, it was found that the 13.5 objective lens was strained. When another 13.5 objective lens was used, the usual four points of maximum intensity were observed. The diagonal positions of the remainder of the samples were checked using both 13.5 lenses and the azimuths coincided, showing no error in this source of the data.

12. The results of corroding specimens A, C, D and F in distilled water at 315° C for 800 hours showed that specimens A, C, and D were of about equal quality while specimen F showed greater corrosion.

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Chapter V

RECOMMENDATIONS FOR FURTHER INVESTIGATION

It is recommended that further study, using polarized light, be made of the characteristics of the corrosion film of zirconium. Some suggestions which may prove beneficial in such a study are listed as follows:

1. Preparation of specimens
   (a) Do all drip-melting in a vacuum of 10^{-6} microns to reduce oxygen pickup in the specimens.
   (b) Since chemical analysis indicated that the largest percentages of miscellaneous impurities were removed by volatilization during the first few meltings, drip melt the mother billet of crystal bar about six times for reduction of impurities and for homogenization. Cut the billet into four parts and add nitrogen so that the specimens to be studied will contain approximately 0, 100, 200 and 500 parts per million of nitrogen. This procedure will give each specimen the same number of drip meltings and will reduce the variation in impurities.
   (c) Make specimens larger and provide an additional 10 grams for chemical analysis plus 5 grams for standard corrosion resistance test.
2. Since the metallograph optical system and its associated lenses are being returned to the factory for correction of optical errors, this problem should not be a factor in future work. However, periodic calibration checks should be made on the metallograph.
3. Instead of using a large single crystal in the specimen for study, it

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is suggested that the specimens consist of a large number of small grains that are given a preferential direction by cold working. By using a large area with small grains, i.e, about 100 grains, the problem may be approached in a statistical manner. This may be done by scribing an area on the specimen and centering this area after each autoclave so that the same area will be observed each time. Additional advantages of this method are that the effects of any preferential corrosion along grain boundaries will be included and any preferential corrosion due to crystal orientation will be eliminated. After any cold working, the specimens should be annealed. Twins in the field being measured give erroneous results.

4. Use the elliptical compensator with an optically inactive standard for locating the diagonal positions as well as for determining the phase shift and rotation in the reflected light. This procedure is outlined in the Bausch and Lomb Elliptical Vibration Compensator Instruction Book.[9]

5. Record intensities of diagonal positions and of minimum positions. In order to do this it will be desirable to make one or more "intensity standards" to which the galvanometer can be calibrated.

6. Autoclave at 295° C for one hour intervals.

7. Carefully note and record the color and general macroscopic surface appearance for each measurement. It is entirely possible that the corrosion time at which a specimen first shows film breakdown (as described in Chapt. IV-8) may be used as a corrosion parameter.

Appendix A

Geometrical Analysis of Poincare Sphere for Phase Shift Due to Specimen

From Figure A-1

$$AB = CA \sin(2m + 2d_1)$$

$$AC = AB \tan \varphi_1 = OA \tan \varphi_1 \sin(2m + 2d_1)$$

$$\tan \delta_1 = \frac{AC}{OA} = \frac{OA \tan \varphi_1 \sin(2m + 2d_1)}{OA}$$

$$\tan \delta_1 = \tan \varphi_1 \sin(2m + 2d_1) \qquad \text{------ (a)}$$

Similarly

$$PR = OP \sin(2m - 2d_2)$$

$$QP = PR \tan \varphi_2 = OP \tan \varphi_2 (\sin 2m - 2d_2)$$

$$\tan \delta_2 = \frac{QP}{OP} = \frac{OP \tan \varphi_2 \sin(2m - 2d_2)}{OP}$$

$$\tan \delta_2 = \tan \varphi_2 \sin(2m - 2d_2) \qquad \text{------ (b)}$$

Since phase shift due to specimen is $\Delta = \delta_1 + \delta_2$

and $\varphi_1 \approx \varphi_2 = \varphi$

adding (a) and (b)

$$\tan \delta_1 + \tan \delta_2 = \tan \varphi \left[\sin(2m + 2d_1) + \sin(2m - 2d_2) \right]$$

since $\tan(\delta_1 + \delta_2) = \dfrac{\tan \delta_1 + \tan \delta_2}{1 - \tan \delta_1 \tan \delta_2}$

and since δ_1 and $\delta_2 \ll 1$

$$\tan(\delta_1 + \delta_2) \approx \tan \delta_1 + \tan \delta_2$$

or $$\tan \Delta = \tan \varphi \left[\sin(2m + 2d_1) + \sin(2m - 2d_2) \right]$$

and Δ and φ are both small so

$$\sin \Delta = \sin \varphi \left[\sin(2m + 2d_1) + \sin(2m - 2d_2) \right]$$

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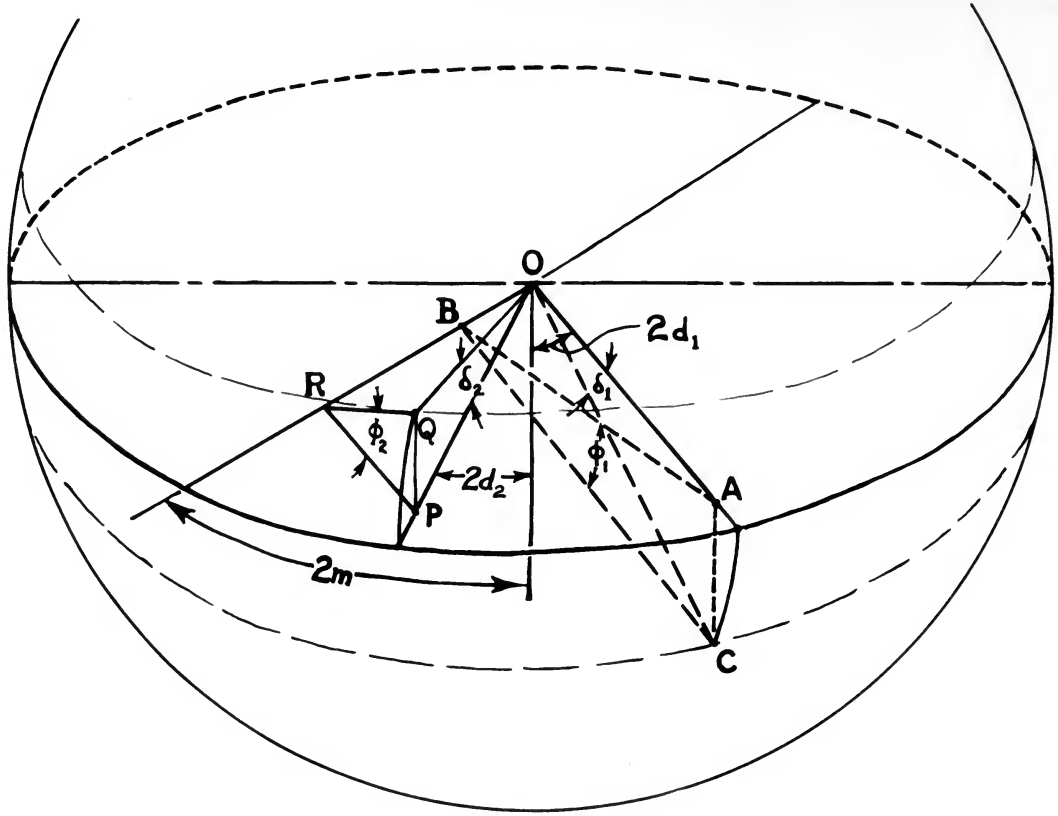

Fig. A-1

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Appendix B

SAMPLE CALCULATION FOR ADDITION OF NITROGEN

Problem: To add 40 ppm nitrogen to a zirconium billet weighing 87.5 gms.

$$\frac{40}{10^6} = \frac{x}{87.5} \quad \text{and} \quad x = \frac{40 \times 87.5}{10^6} = \text{number gms } N_2 \text{ needed}$$

Convert gms. of N_2 to cc of N_2 at STP and convert for room temperature

$$\text{cc of } N_2 = \frac{40 \times 87.5}{10^6} \times \frac{22{,}400}{28} \times \frac{300}{273}$$

Estimate that an excess of 20% N_2 must be supplied because a portion of the available N_2 is not absorbed when the zirconium billet is heated.

$$\therefore \text{cc of } N_2 = \frac{1.2 \times 40 \times 87.5 \times 22{,}400 \times 300}{10^6 \times 28 \times 273} = V_1$$

Volume of manifold = 325 cc = V_2

P_1 = atmos. press. (on this day = 751 mm)

$$P_1V_1 = P_2V_2 \quad \text{or} \quad P_2 = \frac{P_1 V_1}{V_2}$$

$$\therefore P_2 = \frac{751 \times 1.2 \times 40 \times 87.5 \times 22{,}400 \times 300}{10^6 \times 28 \times 273 \times 325} = 8.54 \text{ mm}$$

Consequently, bleed nitrogen from the circular flask (shown at the top of Fig. B-1) into the manifold until the pressure manometer reads 8.54 mm. Open the stopcock to the quartz tube (Fig. III-1) containing the zirconium billet and heat the billet by induction coil to 1600° C for about ten minutes or until the 8.54 mm pressure of N_2 has been reduced essentially to the original vacuum pressure.

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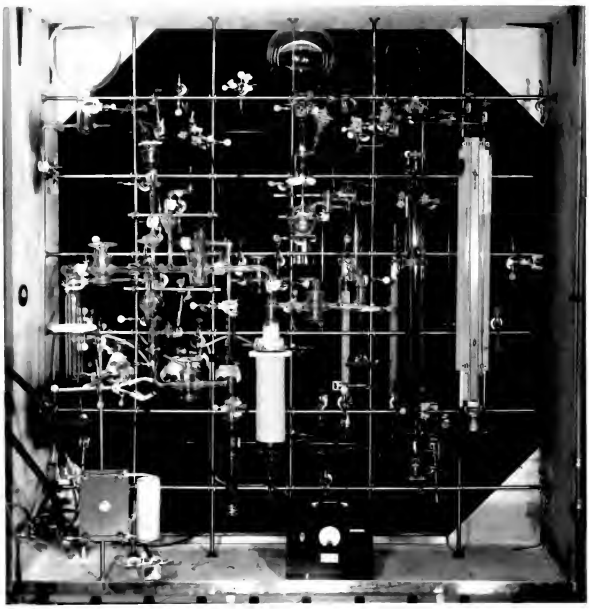

Figure B-1

TYPICAL LABORATORY APPARATUS FOR
ADDING NITROGEN TO ZIRCONIUM

Appendix C

CHEMICAL AND SPECTROGRAPHIC ANALYSIS

Table I gives the results of Chemical and Spectrographic Analysis of the original Westinghouse Grade I Crystal Bar Zirconium and of the six samples (A, B, C, D, E, and F) made from that material. During the nitrogen addition there was no substantial change in concentration of any other elements except oxygen, carbon and iron. Of these, the oxygen increased while the iron decreased. While the small amount of oxygen pickup noticed would not essentially alter one way or other the corrosion resisting qualities of the samples, the reduction in iron content might well have a pronounced effect. In particular, Sample A which was purest in nitrogen was most impure in iron, with a ratio of iron impurity to nitrogen impurity of more than 20:1.

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TABLE C-I

CHEMICAL CONTENT OF PARENT ZIRCONIUM AND

OF SIX SAMPLES MADE THEREFROM

	Chem. # 1	Spect.	Chem. # A	Spect.	Chem. # B	Spect.	Chem. # C	Spect.	Chem. # D	Spect.	Chem. # E	Spect.	Chem. # F	Spect.
C	110		90				140		50		140		80	
O	70		90		260		380		290		450		320	
N	2		6		70		115		147		150		240	
Fe	152												65	
Si	<10-20		30		35		30		20				15	
Al	54-40		45		40		40		30		40		55	
Ti	<5-9		6		μ1		μ1		μ1		μ1		<5	μ1
Ni	6-8		5		μ1		μ1		μ1		μ1		<5	μ1
Cr	<5-65		30		25		20		25		30		<5	20
Ca	12		7		9		8		8		10		8	
Cu	9		5		4		μ1		μ1		2		μ1	
Mg	2		μ1		2		1		μ1		20		5	
Mn	4		μ1		μ1		μ1				μ1		μ1	
Mo	μ10		μ10		μ10		μ10		μ10		μ10		μ10	
Pb	3		μ1		μ1		μ1		μ1		μ1		μ1	
Sn	2		1		μ1		μ1		μ1					
V	μ50		μ50		μ50		μ50		μ50		μ50		μ50	

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Appendix D

Data

The data consists of values of phase shift and of rotation of plane of polarisation measured after electropolishing and after each autoclaving. It was plotted directly on graph paper forms designed especially for this purpose.

For each specimen for each run, a curve was faired through the measured values of 2m and of 2r versus the number of autoclavings. These curves are included at the end of each section of data. The values used in the calculations of Appendix F are values picked off the faired curves of data.

Run #1

Corrosion film was grown at 235° C

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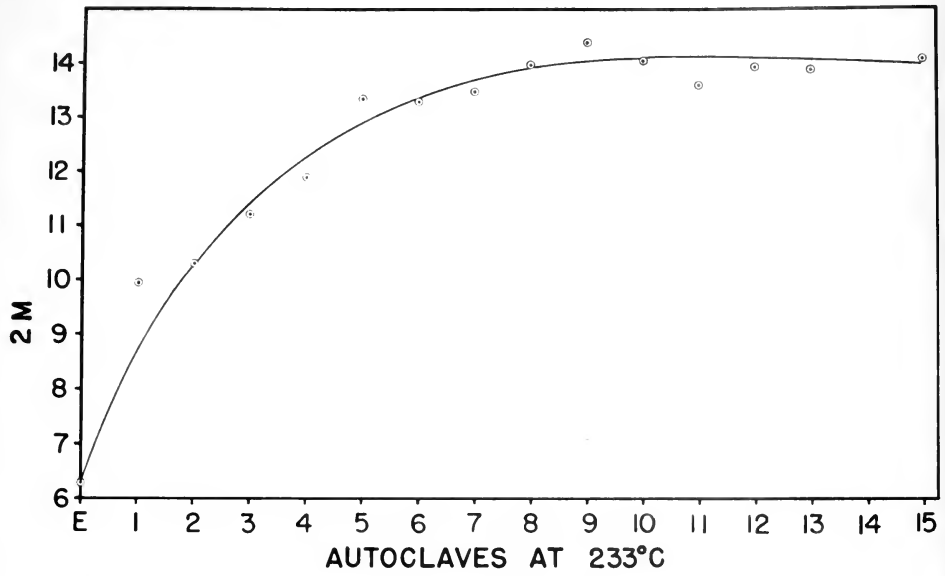
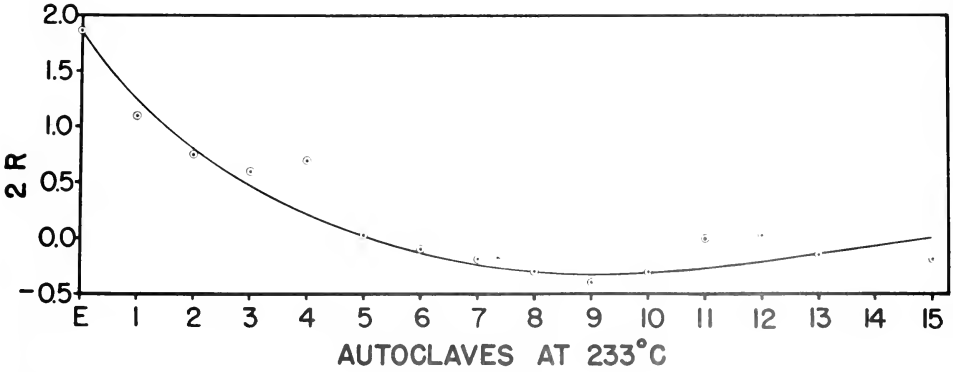




PHASE SHIFT (2m) AND ROTATION OF PLANE OF POLARIZATION (2r) VS. CORROSION TIME FOR SAMPLE NO. 1A-1

SAMPLE No. 1A 6 PPM N_2 DATE 4 March 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION AS ELECTROPOLISHED

@ 080		@ 170		@ 260		@ 350	
M_1	R_1	M_3	R_3	M_3	R_3	M_4	R_4
45.2	46.8	38.9	44.6	45.5	46.2	39.0	44.7
45.3	46.7	39.3	44.5	45.0	46.7	38.6	45.1
Av. 45.25	46.75	39.10	44.55	45.25	46.45	38.80	44.90

M_1	45.25	R_1	46.75
M_2	39.10	R_2	44.55
ΔM_{12}	6.15	ΔR_{12}	2.20

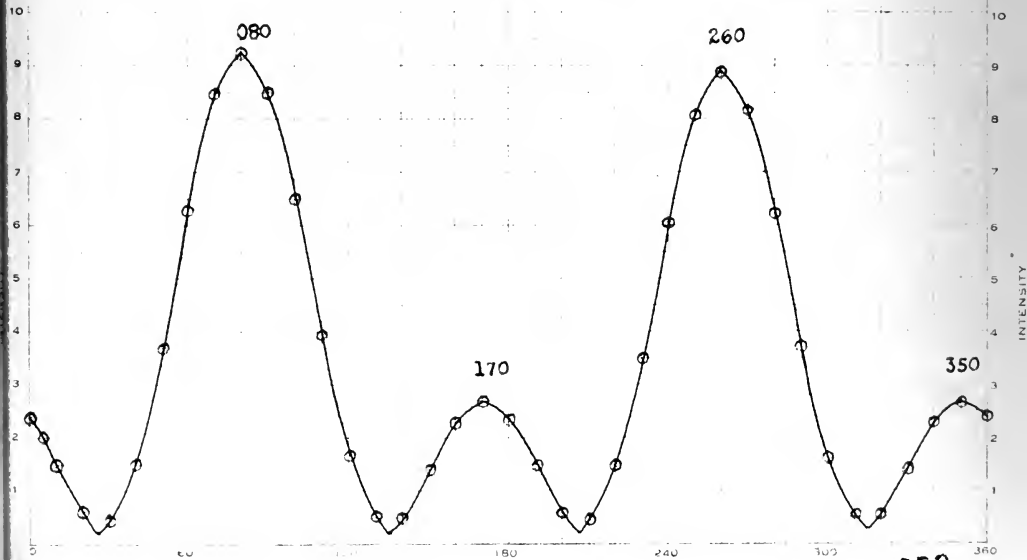
M_3	45.25	R_3	46.45
M_4	38.80	R_4	44.90
ΔM_{34}	6.45	ΔR_{34}	1.55

Av ΔM 6.30 PHASE SHIFT

Av ΔR 1.87 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



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SAMPLE No. 1A 6 PPM N2 DATE 12 March PM

CRYSTAL No 1 SURFACE FILM CONDITION 1st AUTOCLAVE 15 min 233°C

	@ 332		@ 062		@ 152		@ 242	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	47.1	46.1	37.3	45.1	47.0	46.2	37.3	45.4
	47.6	46.4	37.0	45.0	47.0	46.1	37.3	44.9
Av	47.35	46.25	37.15	45.05	47.00	46.15	37.30	45.15

M_1	47.35	R_1	46.25
M_2	37.15	R_2	45.05
ΔM_{12}	10.20	ΔR_{12}	1.20

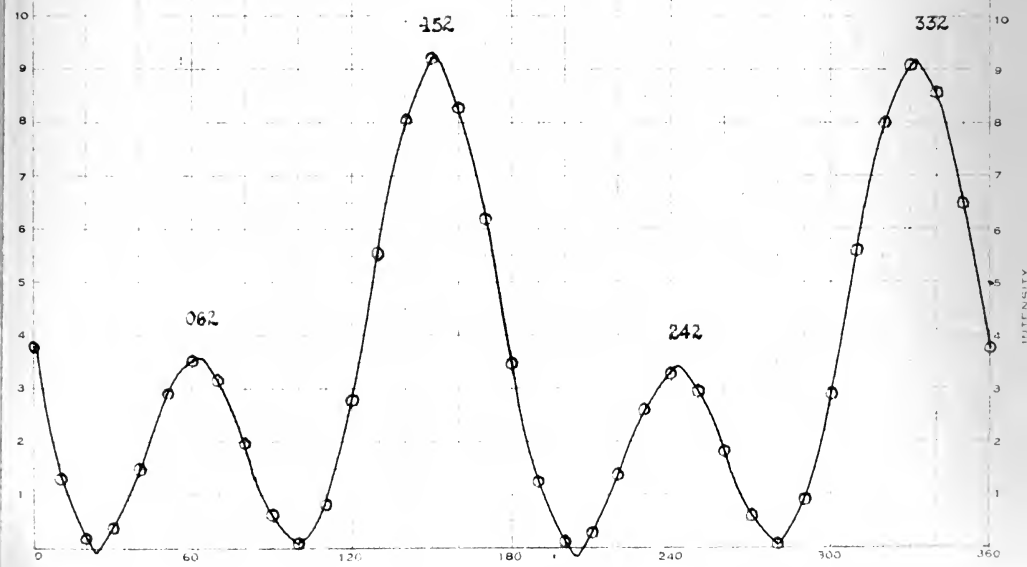
M_3	47.00	R_3	46.15
M_4	37.30	R_4	45.15
ΔM_{34}	9.70	ΔR_{34}	1.00

Av ΔM 9.95 PHASE SHIFT

Av ΔR 1.10 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



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SAMPLE No 1A 6 PPM H_2 DATE 14 MARCH 1952 PM

CRYSTAL No 1 SURFACE FILM CONDITION 2nd AUTOCLAVE OF 15 Min. at 233°C

@ 317		@ 047		@ 137		@ 227	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
47.2	45.7	37.2	44.3	47.9	45.9	37.1	44.7
46.9	45.9	37.4	45.1	47.9	45.8	37.0	45.2
Av. 47.05	45.80	37.30	45.20	47.90	45.85	37.05	44.95

M_1	47.05	R_1	45.80
M_2	37.30	R_2	45.20
ΔM_{12}	9.75	ΔR_{12}	.60

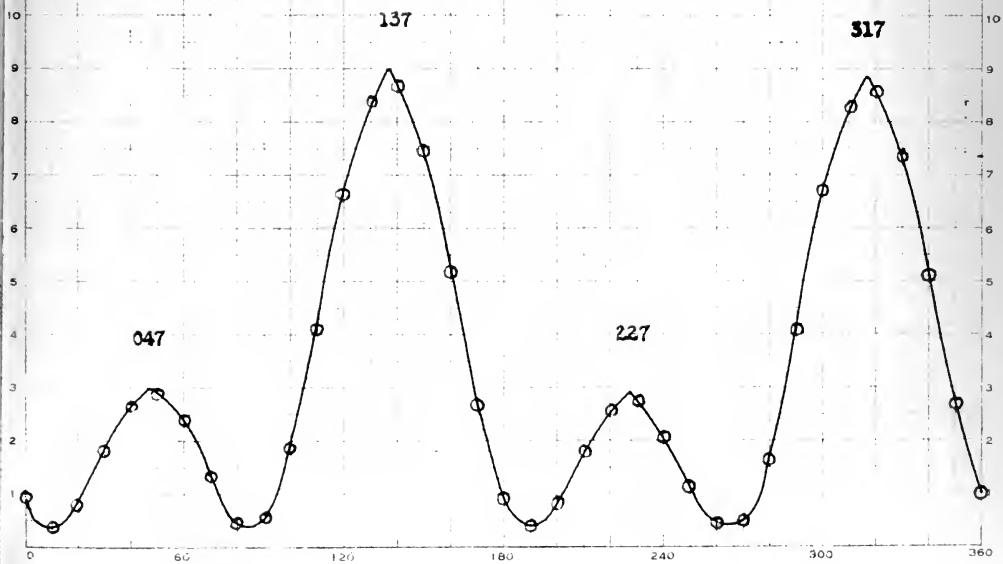
M_3	47.90	R_3	45.85
M_4	37.05	R_4	44.95
ΔM_{34}	10.85	ΔR_{34}	.90

Av ΔM 10.30 PHASE SHIFT

Av ΔR .75 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



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SAMPLE No — PPM DATE

CRYSTAL No 1 SURFACE FILM CONDITION

	@ 068		@ 158		@ 248		@ 338	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	44.2	46.5	34.2	46.7	46.5	46.2	37.3	45.6
	46.1	46.1	34.0	45.4	46.1	46.1	37.3	45.7
Av.	45.50	46.30	34.10	45.50	46.30	46.15	37.30	45.65

M_1	45.50	R_1	46.30
M_2	34.10	R_2	45.90
ΔM_{12}	11.40	ΔR_{12}	.70

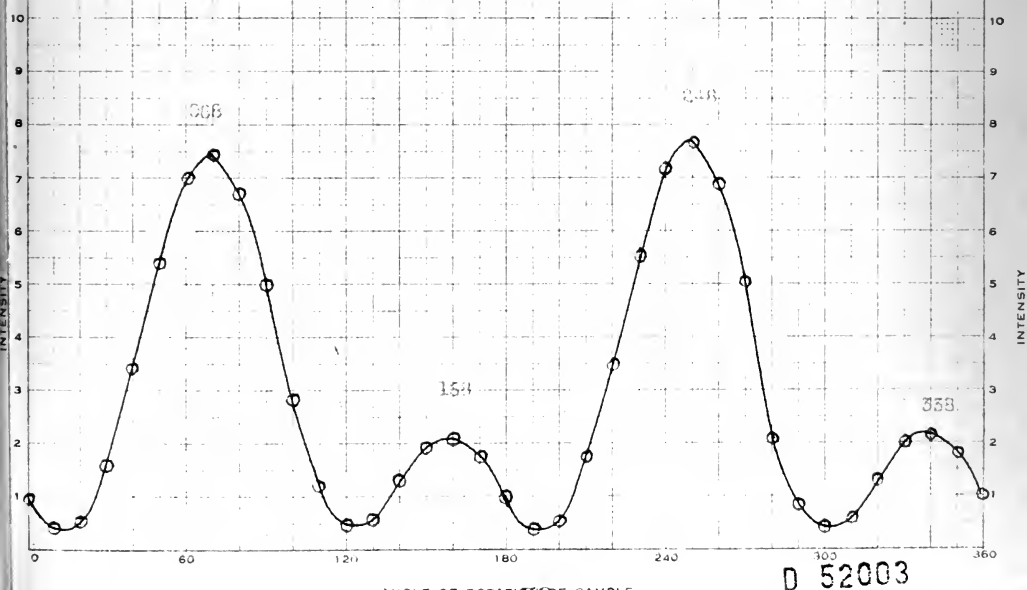
M_3	46.30	R_3	46.15
M_4	37.30	R_4	45.65
ΔM_{34}	11.00	ΔR_{34}	.50

Av ΔM 11.20 PHASE SHIFT

Av ΔR .60 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. [illegible] PPM [illegible] DATE [illegible]

CRYSTAL No 1 SURFACE FILM CONDITION

	@ [illegible]		@ 146		@ [illegible]		@ [illegible]	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	48.2	45.5	56.4	44.6	47.8	45.0	[illegible]	44.9
	48.1	45.5	56.3	44.9	47.8	45.5	[illegible]	[illegible]
AV.	48.15	45.50	36.35	44.75	47.80	45.25	[illegible]	44.25

M_1	42.15	R_1	45.50
M_2	36.35	R_2	44.75
ΔM_{12}	11.80	ΔR_{12}	.80

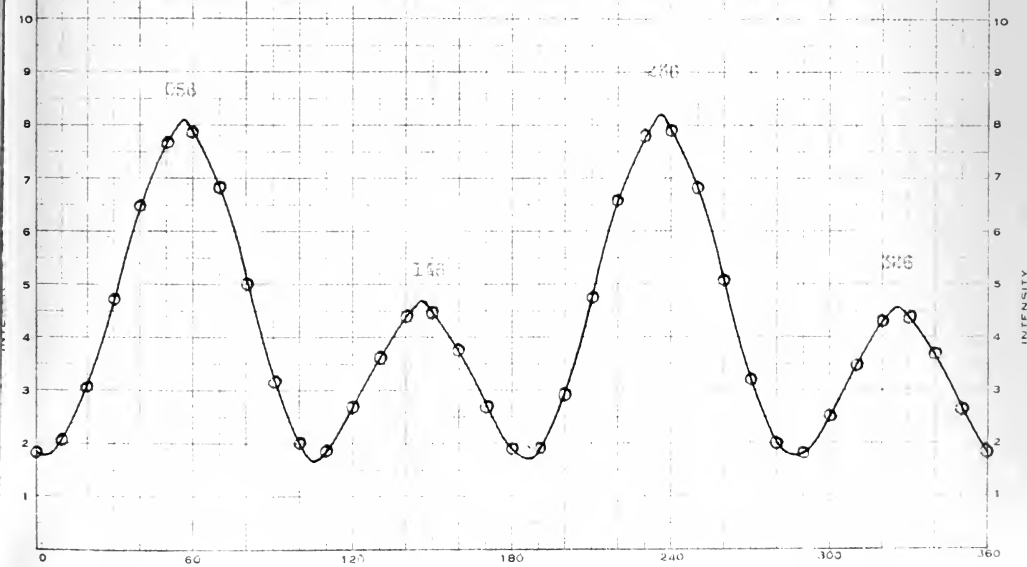
M_3	47.80	R_3	45.75
M_4	[illegible]	R_4	44.75
ΔM_{34}	11.35	ΔR_{34}	.80

Av ΔM 11.[illegible] PHASE SHIFT

Av ΔR .70 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



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SAMPLE No [illegible] PPM [illegible] DATE [illegible]

CRYSTAL No [illegible] SURFACE FILM CONDITION [illegible]

@ [illegible]		@ [illegible]		@ 135		@ [illegible]	
M	R	M_2	R_2	M_3	R_3	M_4	R_4
48.1	[illegible]	[illegible]	[illegible]	47.9	45.4	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
Av. [illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

M_1 [illegible] R_1 [illegible]

M_2 [illegible] R_2 [illegible]

ΔM_{12} 13.20 ΔR_{12} [illegible]

M_3 47.35 R_3 46.45

M_4 [illegible] R_4 [illegible]

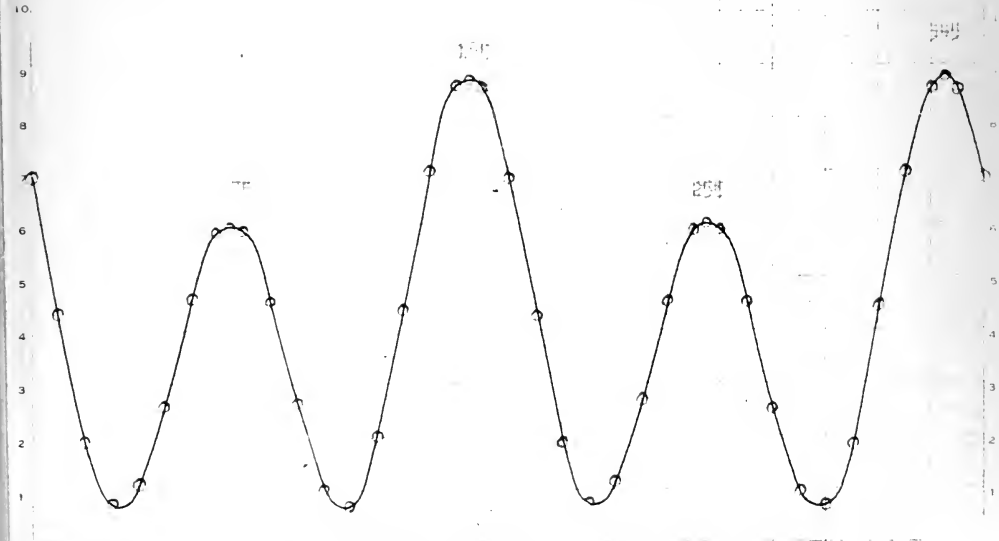
ΔM_{34} [illegible] ΔR_{34} .05

Av ΔM [illegible] PHASE SHIFT

Av ΔR .03 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 1A PPM [illegible] DATE [illegible]

CRYSTAL No 2 SURFACE FILM CONDITION [illegible]

@ 35°		@ 25°		@ 15°		@ [illegible]	
M1	R1	M2	R2	M3	R3	M4	R4
49.4	45.2	35.7	45.4	49.6	45.2	36.4	45.5
49.0	45.4	36.0	45.5	49.4	45.3	36.2	45.3
Av. 49.20	45.30	35.85	45.45	49.50	45.25	36.30	45.40

M_1	49.20	R_1	45.30
M_2	35.85	R_2	45.45
ΔM_{12}	13.35	ΔR_{12}	- .15

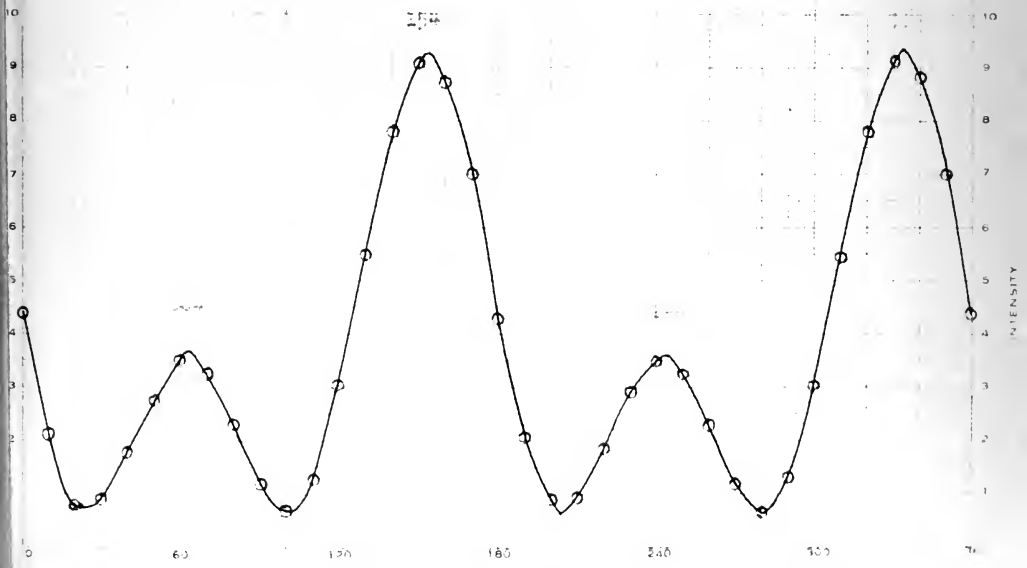
M_3	49.50	R_3	45.25
M_4	36.30	R_4	45.40
ΔM_{34}	13.20	ΔR_{34}	- .05

Av ΔM 13.28 PHASE SHIFT

Av ΔR - .10 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

D 52003

SAMPLE No 1A 5 PPM I_2 DATE 20 MARCH 1[illegible]

CRYSTAL No 1 SURFACE FILM CONDITION 7th AUTOCLAVE OF 15 MIN @ 233°C

@ 090		@ 180		@ 270		@ 360	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
51.1	45.7	37.8	45.8	50.8	45.3	37.1	45.7
51.1	45.7	37.9	45.8	51.0	45.6	37.3	45.7
51.10	45.70	37.85	45.80	50.90	45.45	37.20	45.70

M_1 51.10 R_1 45.70
M_2 37.85 R_2 45.80
ΔM_{12} 13.25 ΔR_{12} - .10

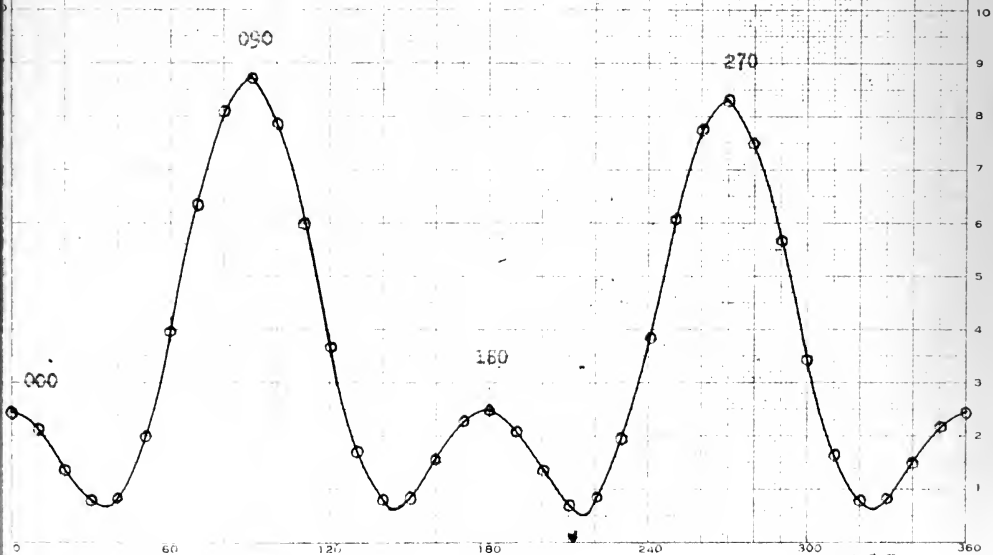
M_3 50.90 R_3 45.45
M_4 37.20 R_4 45.70
ΔM_{34} 13.70 ΔR_{34} - .25

Av ΔM 13.48 PHASE SHIFT

Av ΔR - .18 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



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SAMPLE No. 1A 6 PPM N_2 DATE 27 MARCH 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 8th AUTOCLAVE OF 15 MIN. @ 233°C

@ 335		@ 065		@ 155		@ 245	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
50.2	45.2	35.9	45.7	49.8	45.6	35.7	46.0
50.0	45.7	36.1	45.5	49.6	45.5	36.0	46.0
Av. 50.10	45.45	36.00	45.60	49.70	45.55	35.85	46.00

M_1	50.10	R_1	45.45
M_2	36.00	R_2	45.60
ΔM_{12}	14.10	ΔR_{12}	- .15

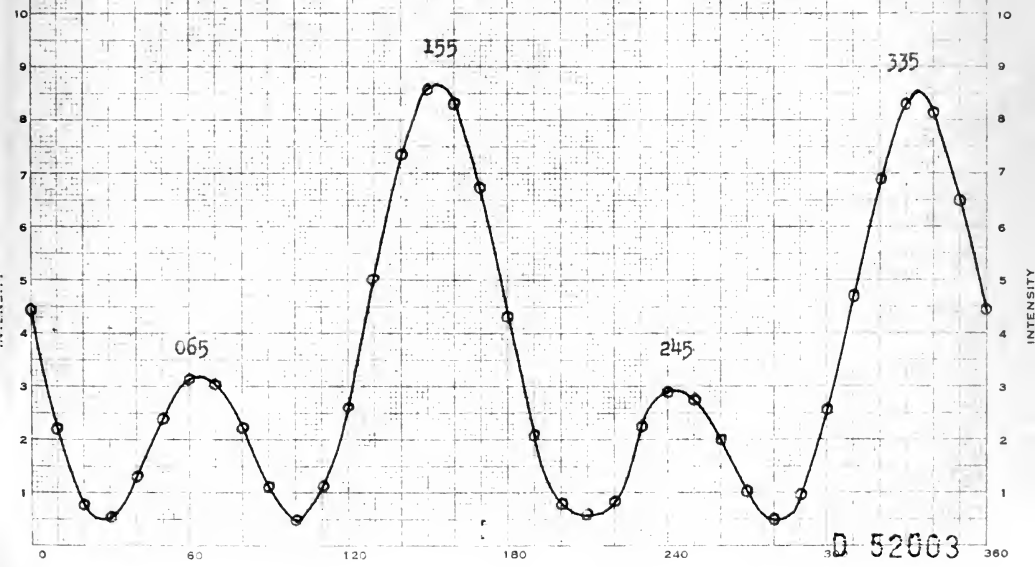
M_3	49.70	R_3	45.55
M_4	35.85	R_4	46.00
ΔM_{34}	13.85	ΔR_{34}	- .45

Av ΔM 13.98 PHASE SHIFT

Av ΔR - .30 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

D 52003

SAMPLE No 1A 6 PPM N_2 DATE 29 MARCH 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 9th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 299		@ 029		@ 119		@ 209	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	51.3	44.8	36.6	45.5	50.6	45.3	36.3	45.6
	51.6	45.3	37.0	45.6	50.6	45.1	36.6	45.4
AV.	51.45	45.05	36.80	45.55	50.60	45.20	36.45	45.50

M_1 51.45 R_1 45.05
M_2 36.80 R_2 45.55
ΔM_{12} 14.65 ΔR_{12} - .50

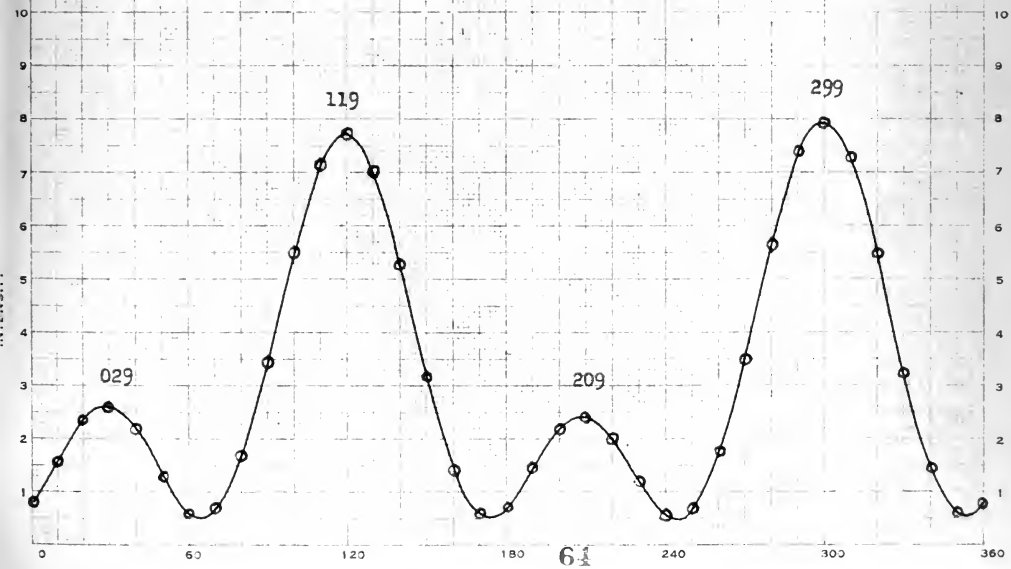
M_3 50.60 R_3 45.20
M_4 36.45 R_4 45.50
ΔM_{34} 14.15 ΔR_{34} - .30

Av ΔM 14.40 PHASE SHIFT

Av ΔR - .40 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1A 6 PPM N_2 DATE 30 MARCH 1952 PM

CRYSTAL No 1 SURFACE FILM CONDITION 10th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 314		@ 044		@ 134		@ 224	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	50.9	45.2	36.8	45.8	50.2	45.5	36.0	45.8
	50.7	45.5	37.0	45.7	50.6	45.8	36.4	45.9
Av.	50.80	45.35	36.90	45.75	50.40	45.65	36.20	45.85

M_1	50.80	R_1	45.35
M_2	36.90	R_2	45.75
ΔM_{12}	13.90	ΔR_{12}	-.40

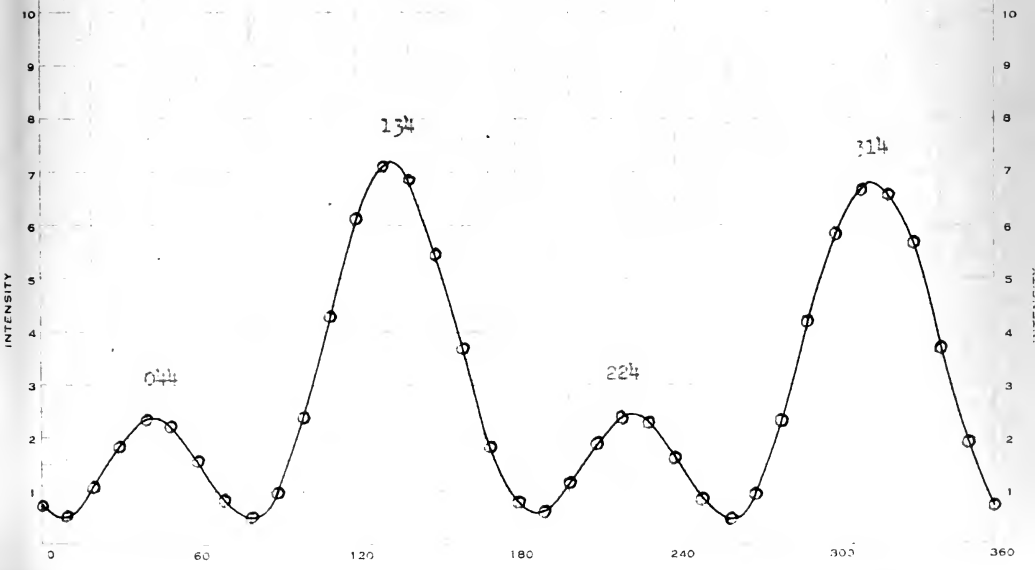
M_3	50.40	R_3	45.65
M_4	36.20	R_4	45.85
ΔM_{34}	14.20	ΔR_{34}	-.20

Av ΔM 14.05 PHASE SHIFT

Av ΔR -.30 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 1A 6 PPM N_2 DATE 1 APRIL 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 11th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 325		@ 055		@ 145		@ 235	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	48.8	45.5	35.3	45.4	48.5	45.5	34.7	45.4
	48.6	45.4	35.3	45.6	48.6	45.5	34.8	45.5
Av.	48.70	45.45	35.30	45.50	48.55	45.50	34.75	45.45

M_1	48.70	R_1	45.45
M_2	35.30	R_2	45.50
ΔM_{12}	13.40	ΔR_{12}	- .05

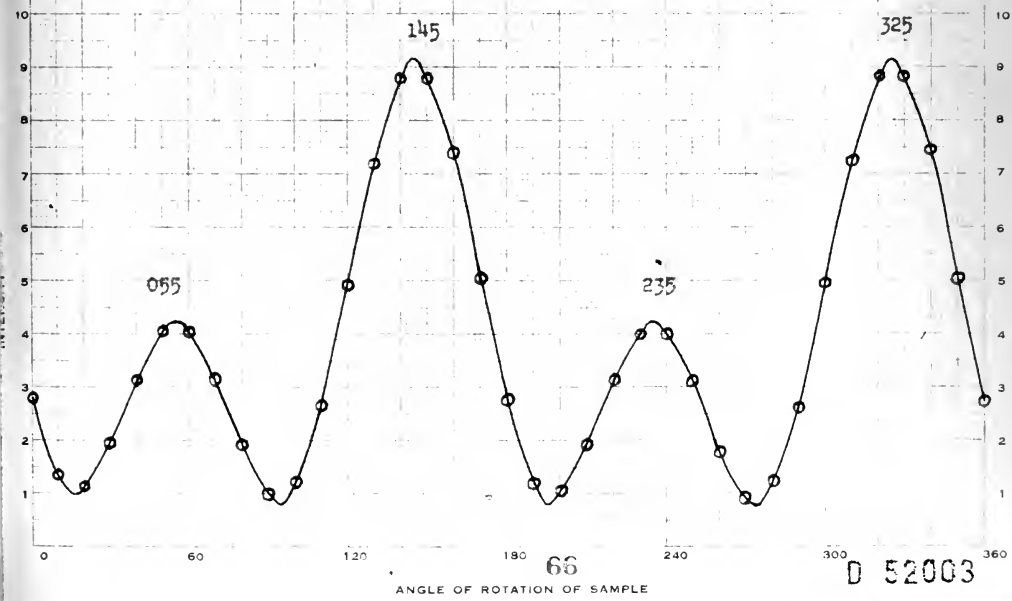
M_3	48.55	R_3	45.50
M_4	34.75	R_4	45.45
ΔM_{34}	13.80	ΔR_{34}	+.05

Av ΔM 13.60 PHASE SHIFT

Av ΔR .00 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No [illegible] PPM [illegible] DATE [illegible]

CRYSTAL No [illegible] SURFACE FILM CONDITION [illegible]

	@ 026		@ 116		@ 206		@ 296	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	49.3	45.6	35.5	45.5	49.5	45.5	35.1	45.4
	49.4	45.5	35.8	45.5	49.0	45.5	35.5	45.5
Av.	49.60	45.55	35.65	45.55	49.25	45.50	35.35	45.45

M_1	49.60	R	45.55
M_2	35.65	R_2	45.55
ΔM_{12}	13.95	ΔR_{12}	.00

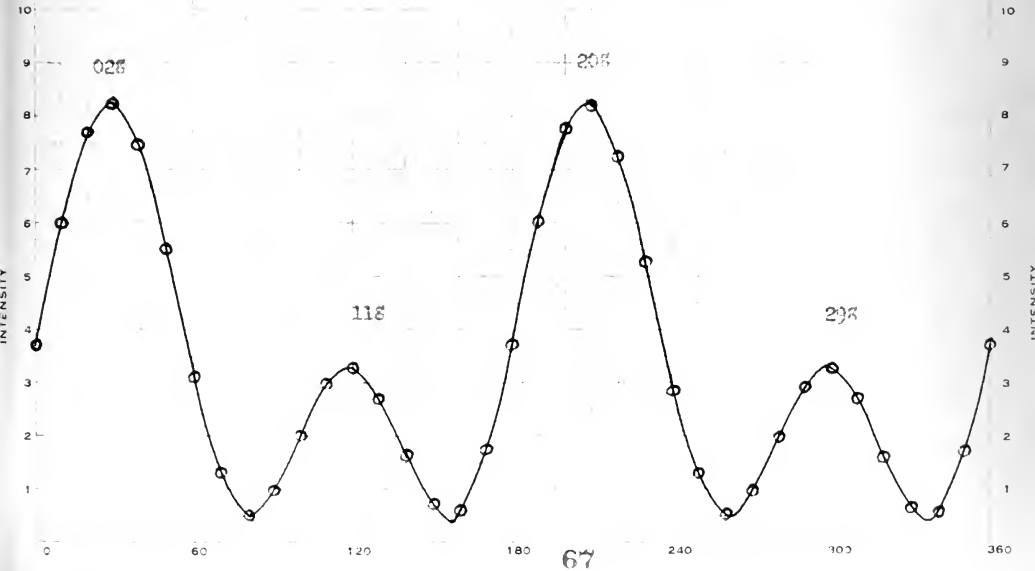
M_3	49.25	R_3	45.50
M_4	35.35	R_4	45.45
ΔM_{34}	13.90	ΔR_{34}	.05

Av ΔM 13.93 PHASE SHIFT

Av ΔR .03 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1A 6 PPM N_2 DATE 3 APRIL 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 13th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 328		@ 058		@ 148		@ 238	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	48.8	45.7	35.3	46.0	49.0	45.5	35.0	45.7
	49.2	45.6	35.3	45.6	48.9	45.7	34.8	45.8
Av.	49.00	45.65	35.30	45.80	48.95	45.60	34.90	45.75

M_1	49.00	R_1	45.65
M_2	35.30	R_2	45.80
ΔM_{12}	13.70	ΔR_{12}	- .15

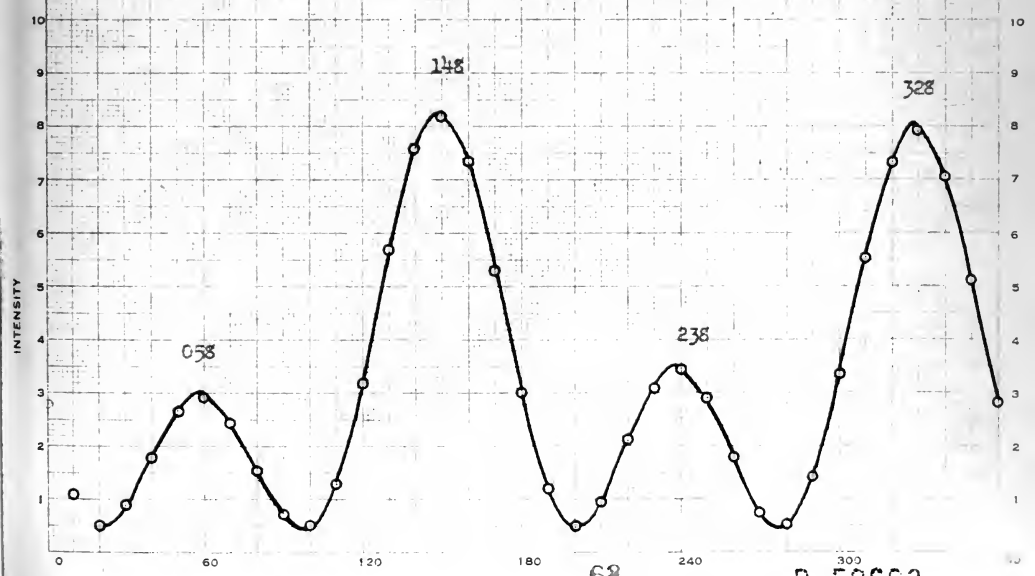
M_3	48.95	R_3	45.60
M_4	34.90	R_4	45.75
ΔM_{34}	14.05	ΔR_{31}	- .15

Av ΔM 13.88 PHASE SHIFT

Av ΔR -.15 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1A 6 PPM H_2 DATE 5 APRIL 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 15th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 150		@ 240		@ 330		@ 060	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	51.1	45.6	36.8	45.8	50.2	45.6	36.6	45.8
	51.1	45.6	36.7	45.7	50.6	45.7	36.6	45.9
Av.	51.10	45.60	36.75	45.75	50.40	45.65	36.60	45.85

M_1 51.10 R_1 45.60

M_2 36.75 R_2 45.75

ΔM_{12} 14.35 ΔR_{12} - .15

M_3 50.40 R_3 45.65

M_4 36.60 R_4 45.85

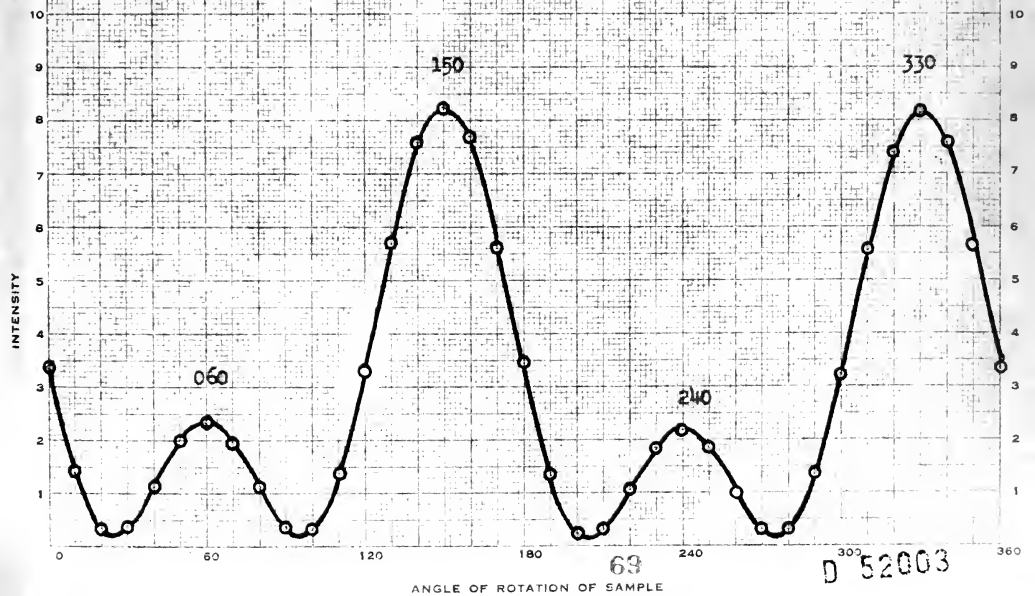
ΔM_{34} 13.80 ΔR_{34} - .20

Av ΔM 14.08 PHASE SHIFT

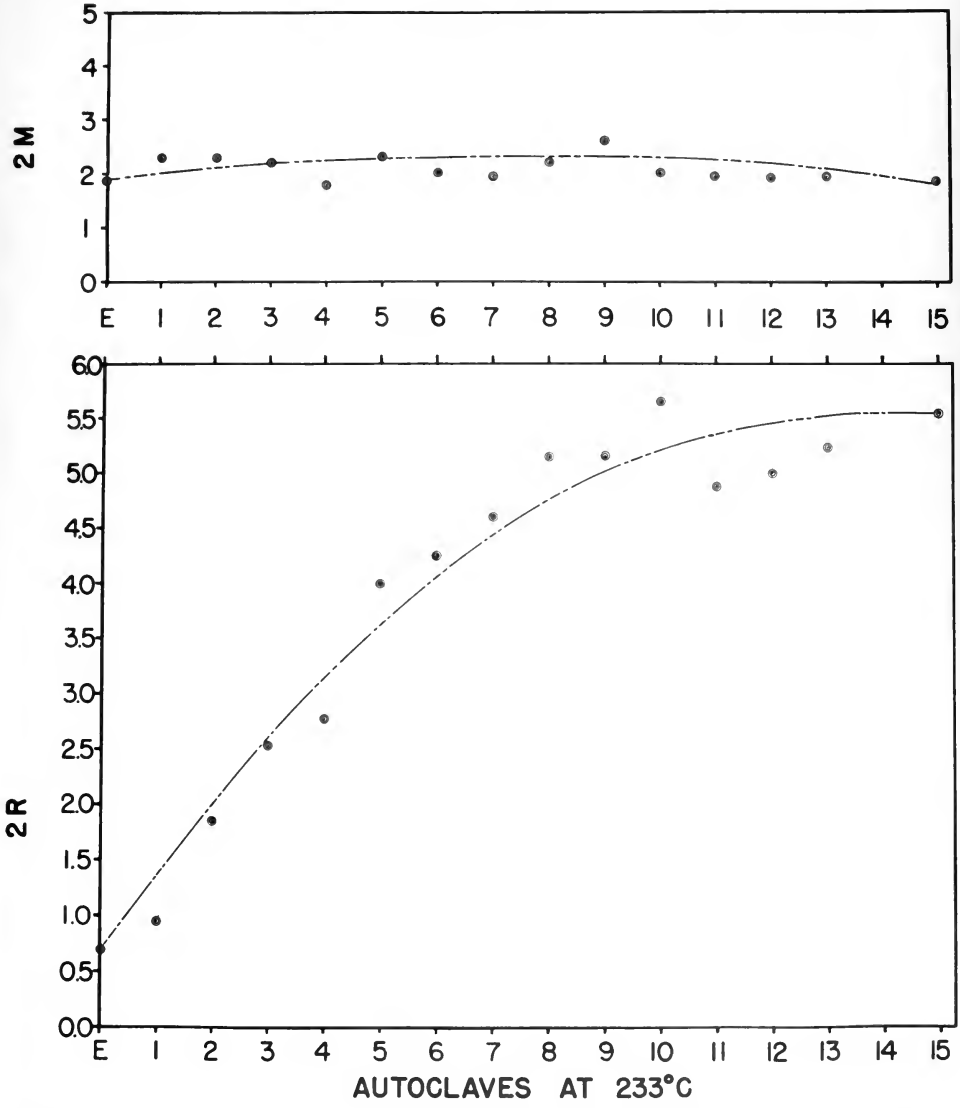
Av ΔR - .18 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



PHASE SHIFT (2m) AND ROTATION OF PLANE OF POLARIZATION (2r) VS. CORROSION TIME FOR SAMPLE NO. 1A-2

SAMPLE No. 1A 6 PPM N_2 DATE 6 March 1952 PM

CRYSTAL No 2 SURFACE FILM CONDITION AS ELECTROPOLISHED

	@ 324		@ 052		@ 144		@ 232	
	M1	R1	M2	R2	M3	R3	M4	R4
	43.5	45.9	42.0	45.0	43.7	45.8	41.6	45.3
	43.6	45.6	41.6	45.1	43.6	46.0	41.7	45.1
Av.	43.55	45.75	41.80	45.05	43.65	45.90	41.65	45.20

M_1	43.55	R_1	45.75
M_2	41.80	R_2	45.05
ΔM_{12}	1.75	ΔR_{12}	.70

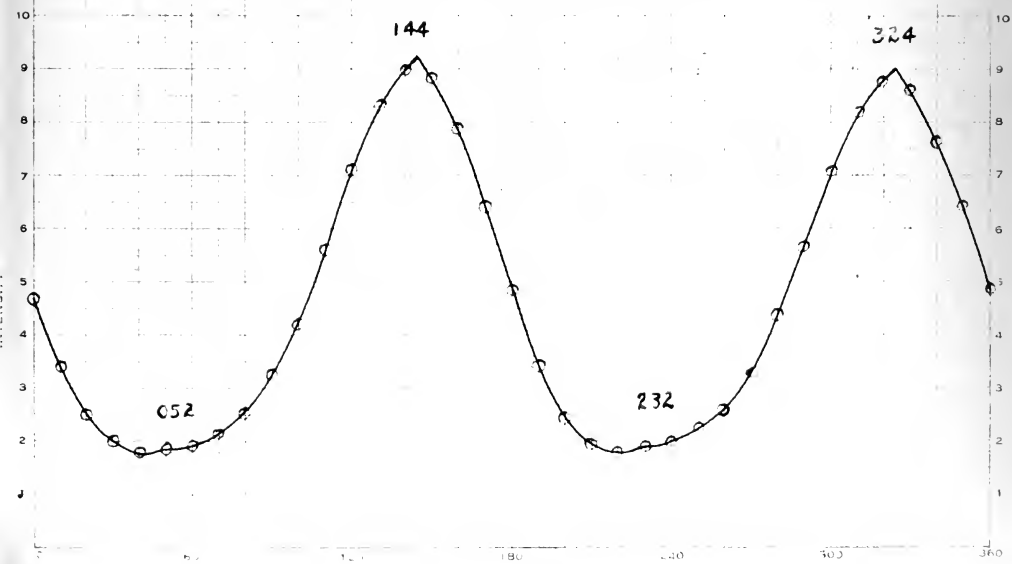
M_3	43.65	R_3	45.90
M_4	41.65	R_4	45.20
ΔM_{34}	2.00	ΔR_{34}	0.70

Av ΔM 1.88 PHASE SHIFT

Av ΔR .70 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 1A 6 PPM N_2 DATE 12 March 1952

CRYSTAL No 2 SURFACE FILM CONDITION 1st AUTOCLAVE 15 Min. at 233°C

	@ 357		@ 087		@ 177		@ 277	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	43.2	45.8	41.2	45.4	44.0	46.8	41.0	45.2
	43.3	46.3	41.0	45.5	43.3	46.7	41.4	45.7
Av	43.25	46.05	41.10	45.45	43.65	46.75	41.20	45.45

M_1	43.25	R_1	46.05
M_2	41.10	R_2	45.45
ΔM_{12}	2.15	ΔR_{12}	0.60

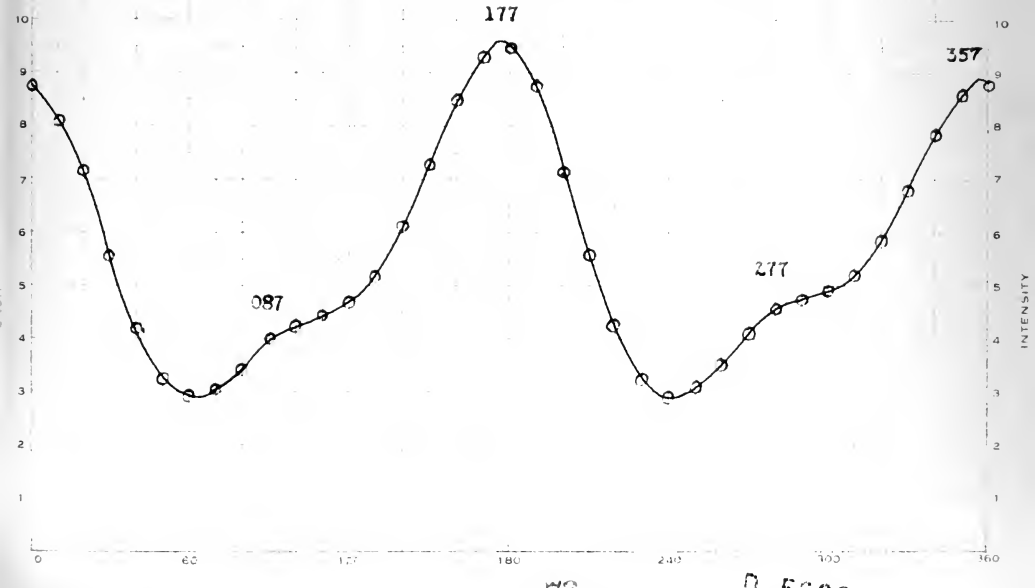
M_3	43.65	R_3	46.75
M_4	41.20	R_4	45.45
ΔM_{34}	2.45	ΔR_{34}	1.30

Av ΔM 2.30 PHASE SHIFT

Av ΔR 0.95 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. [illegible] PPM [illegible] DATE [illegible]

CRYSTAL No. 2 SURFACE FILM CONDITION [illegible]

	@ 026		@ 116		@ 206		@ 296	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	43.6	46.7	41.0	44.6	43.7	45.3	41.6	44.8
	44.0	46.6	41.4	44.6	43.5	45.4	41.6	44.8
Av.	43.80	46.65	41.20	44.60	43.60	45.35	41.60	44.80

M_1	43.80	R_1	46.65
M_2	41.20	R_2	44.60
ΔM_{12}	2.60	ΔR_{12}	2.05

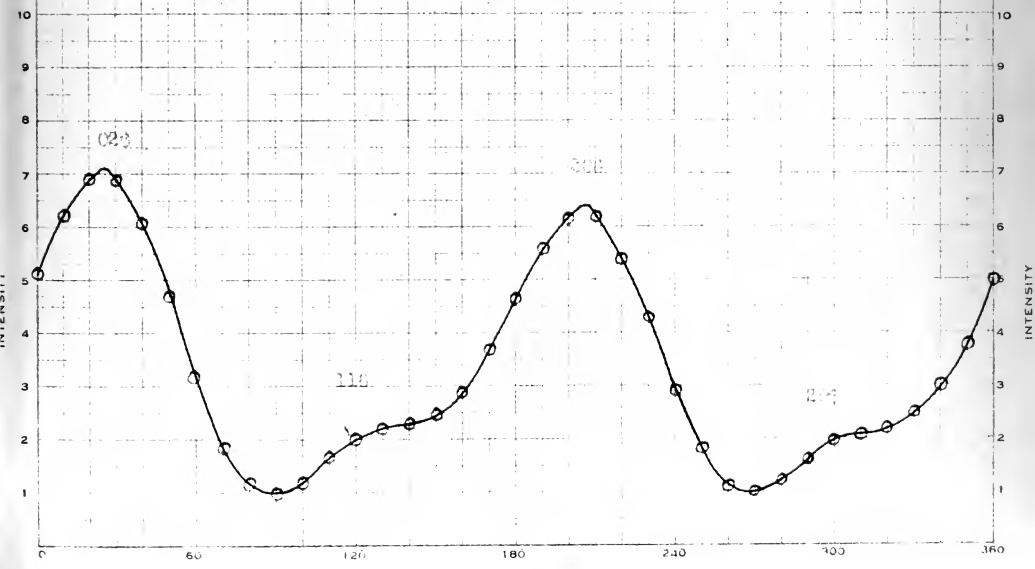
M_3	43.60	R_3	45.35
M_4	41.60	R_4	44.80
ΔM_{34}	2.00	ΔR_{34}	1.55

Av ΔM 2.30 PHASE SHIFT

Av ΔR 1.85 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. [illegible] 0 PPM [illegible] DATE [illegible]

CRYSTAL No. [illegible] SURFACE FILM CONDITION [illegible]

	@ 317		@ 047		@ 137		@ 227	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	46.5	46.7	42.3	44.1	44.1	46.8	42.2	44.1
	44.3	46.6	42.2	44.0	44.3	46.6	41.7	44.4
Av.	46.40	46.65	42.25	44.05	44.20	46.70	41.95	44.25

M_1	44.40	R_1	46.65
M_2	42.25	R_2	44.05
ΔM_{12}	2.15	ΔR_{12}	2.60

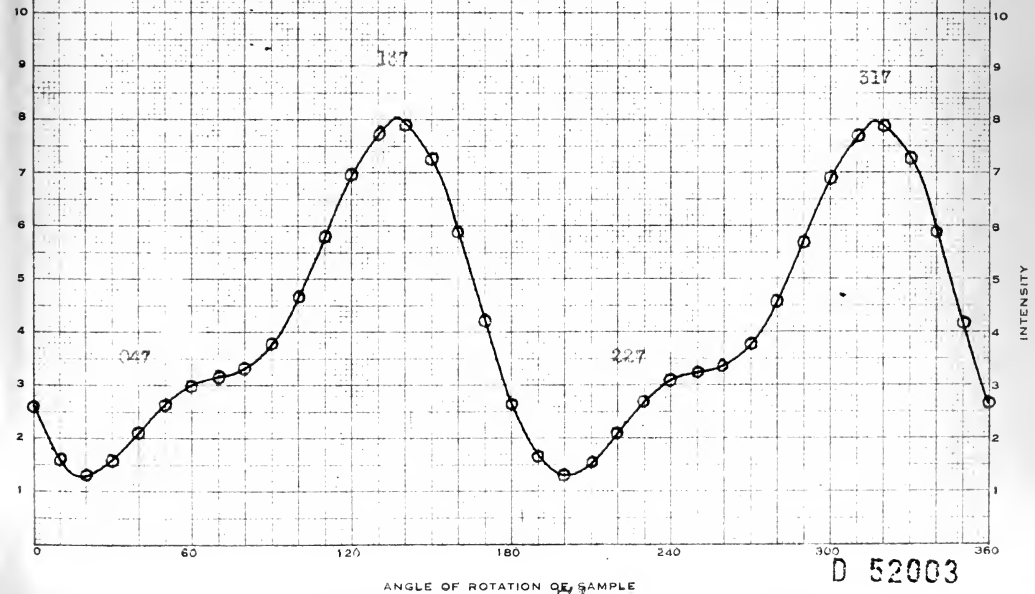
M_3	44.20	R_3	46.70
M_4	41.95	R_4	44.25
ΔM_{34}	2.25	ΔR_{34}	2.45

Av ΔM 2.20 PHASE SHIFT

Av ΔR 2.53 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1A 6 PPM N_2 DATE 22 MARCH 1952 PM

CRYSTAL No. 2 SURFACE FILM CONDITION 4th AUTOCLAVE OF 15 MIN. at 233°

	@ 265		@ 004		@ 086		@ 184	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	42.8	47.0	41.1	43.9	42.2	46.7	40.5	44.2
	42.8	47.0	40.8	44.1	42.4	46.7	40.7	44.1
Av.	42.80	47.00	40.95	44.00	42.30	46.70	40.60	44.15

M_1	42.80	R_1	47.00
M_2	40.95	R_2	44.00
ΔM_{12}	1.85	ΔR_{12}	3.00

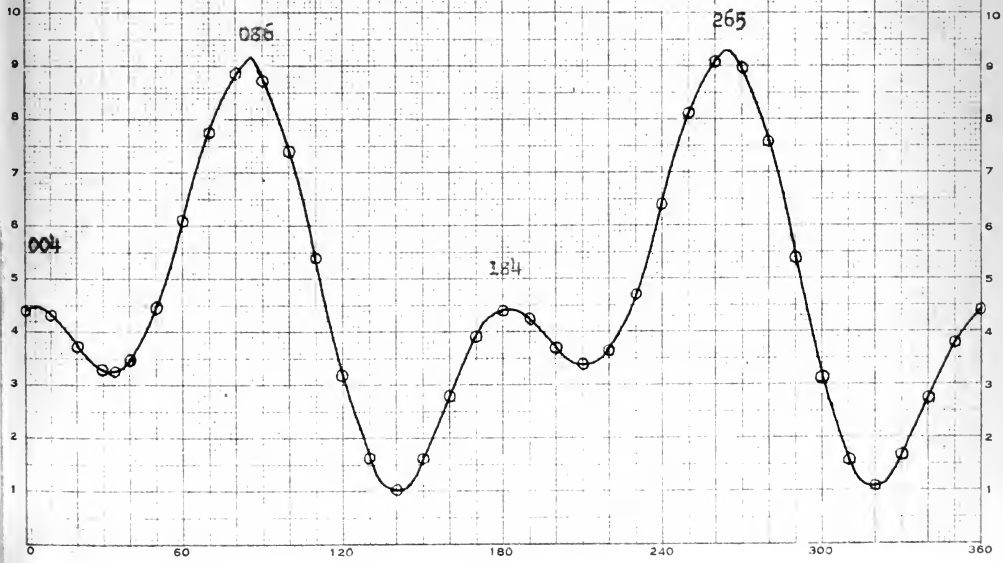
M_3	42.30	R_3	46.70
M_4	40.60	R_4	44.15
ΔM_{34}	1.70	ΔR_{21}	2.55

Av ΔM 1.78 PHASE SHIFT

Av ΔR 2.78 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No [illegible] PPM 2 DATE [illegible]

CRYSTAL No 2 SURFACE FILM CONDITION 5th [illegible]

@ 238		@ 334		@ 058		@ 153	
M_1	R_1	M_4	R_4	M_3	R_3	M_4	R_4
43.2	47.4	40.9	43.5	42.6	47.7	40.5	43.8
43.0	47.4	40.6	43.4	42.8	47.6	40.4	43.4
Av. 43.10	47.40	40.75	43.45	42.70	47.65	40.45	43.60

M_1	43.10	R_1	47.40
M_2	40.75	R_2	43.45
ΔM_{12}	2.35	ΔR_{12}	3.95

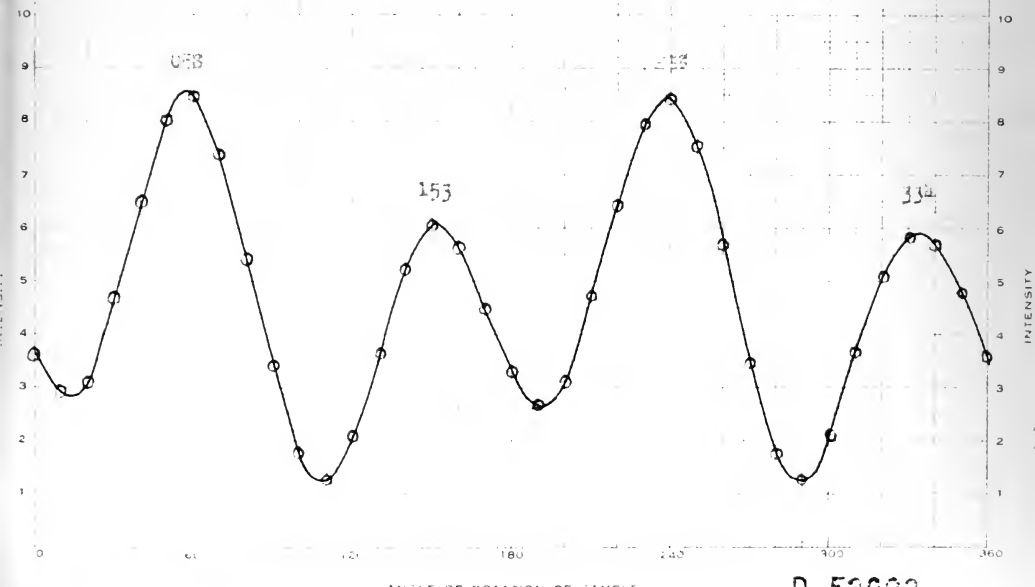
M_3	42.70	R_3	47.65
M_4	40.45	R_4	43.60
ΔM_{34}	2.25	ΔR_{34}	4.05

Av ΔM 2.30 PHASE SHIFT

Av ΔR 4.00 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 1A 6 PPM [illegible] DATE [illegible]

CRYSTAL No [illegible] SURFACE FILM CONDITION [illegible]

	@ 245		@ 248		@ 225		@ [illegible]	
	M_1	R	M_2	R_2	M_3	R_3	M_4	R_4
	44.2	47.8	42.1	43.7	44.0	47.8	42.2	47.6
	44.3	47.8	42.1	43.5	43.9	47.8	42.0	43.3
Av	44.25	47.80	42.1	43.60	43.95	47.80	42.10	43.50

M_1	44.25	R_1	47.80
M_2	42.10	R_2	43.60
ΔM_{12}	2.15	ΔR_{12}	4.20

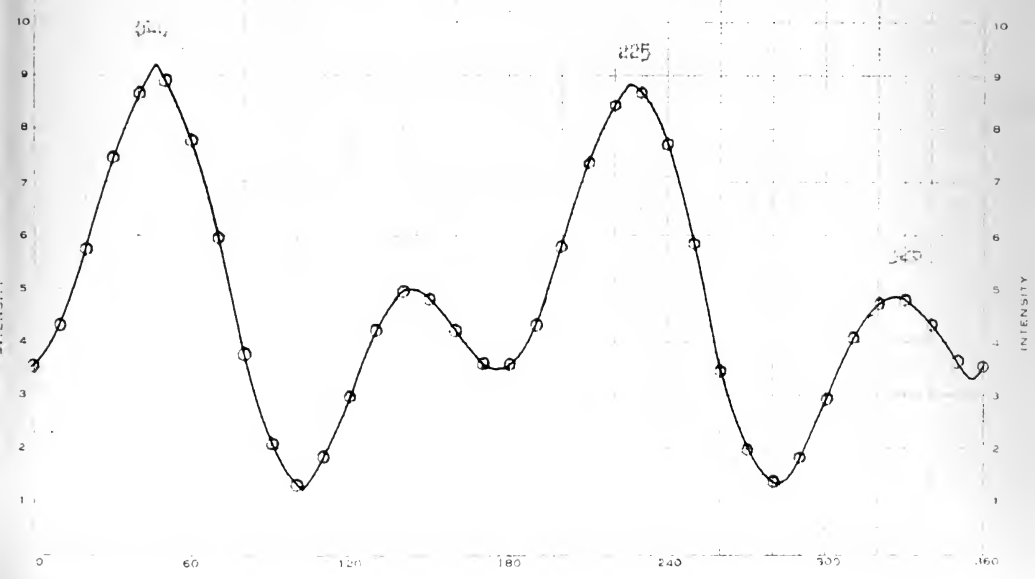
M_3	43.95	R_3	47.80
M_4	42.10	R_4	43.50
ΔM_{34}	1.85	ΔR_{34}	4.30

Av ΔM 2.00 PHASE SHIFT

Av ΔR 4.25 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1A b PPM N_2 DATE 25 MARCH 1952 AM

CRYSTAL No. 2 SURFACE FILM CONDITION 7th AUTOCLAVE OF 15 MIN. @ 233°C

@ 341		@ 083		@ 164		@ 261	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
44.8	47.7	43.0	43.0	44.4	47.6	42.3	43.1
44.7	47.7	42.7	43.2	44.2	47.6	42.4	42.9
Av. 44.75	47.70	42.85	43.10	44.30	47.60	42.35	43.00

M_1	44.75	R_1	47.70
M_2	42.85	R_2	43.10
ΔM_{12}	1.90	ΔR_{12}	4.60

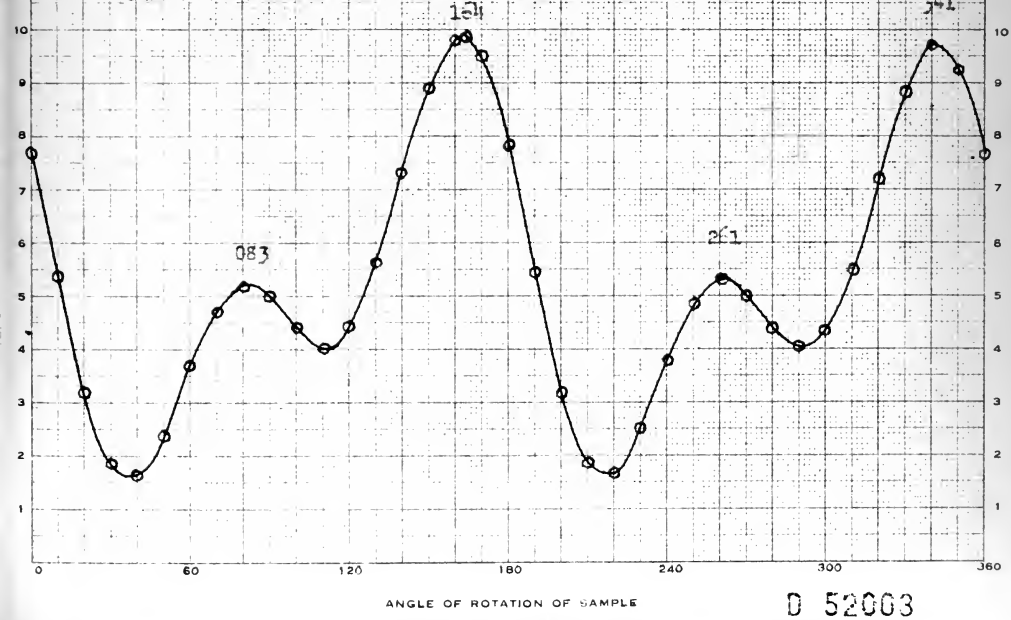
M_3	44.30	R_3	47.60
M_4	42.35	R_4	43.00
ΔM_{34}	1.95	ΔR_{34}	4.60

Av ΔM 1.93 PHASE SHIFT

Av ΔR 4.60 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 1A 6 PPM H_2S DATE 27 MARCH 1952 PM

CRYSTAL No 2 SURFACE FILM CONDITION 8th AUTOCLAVE OF 15 MIN. @ 235°C

	@ 047		@ 145		@ 227		@ 325	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	44.7	48.2	42.0	43.4	44.3	48.4	42.3	42.9
	44.6	48.3	42.4	43.2	44.3	48.2	42.4	43.0
Av.	44.65	48.25	42.20	43.30	44.30	48.30	42.35	42.95

M_1	44.65	R_1	48.25
M_2	42.20	R_2	43.30
ΔM_{12}	2.45	ΔR_{12}	4.95

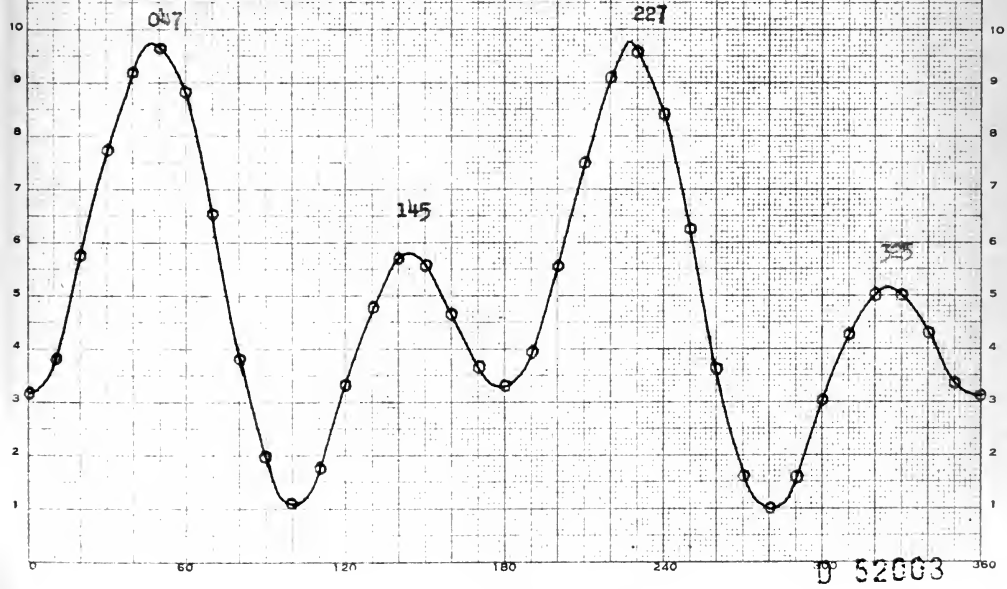
M_3	44.30	R_3	48.30
M_4	42.35	R_4	42.95
ΔM_{34}	1.95	ΔR_{34}	5.35

Av ΔM 2.20 PHASE SHIFT

Av ΔR 5.15 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 1A 6 PPM N_2 DATE 29 MARCH 1952 PM

CRYSTAL No 2 SURFACE FILM CONDITION 9TH AUTOCLAVE OF 15 MIN. @ 235°C

	@ 011		@ 110		@ 192		@ 289	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	45.3	48.3	42.5	43.0	45.2	48.1	42.5	42.9
	45.5	48.2	42.5	43.1	44.7	48.0	42.8	43.0
Av.	45.40	48.25	42.50	43.05	44.95	48.05	42.65	42.95

M_1	45.40	R_1	48.25
M_2	42.50	R_2	43.05
ΔM_{12}	2.90	ΔR_{12}	5.20

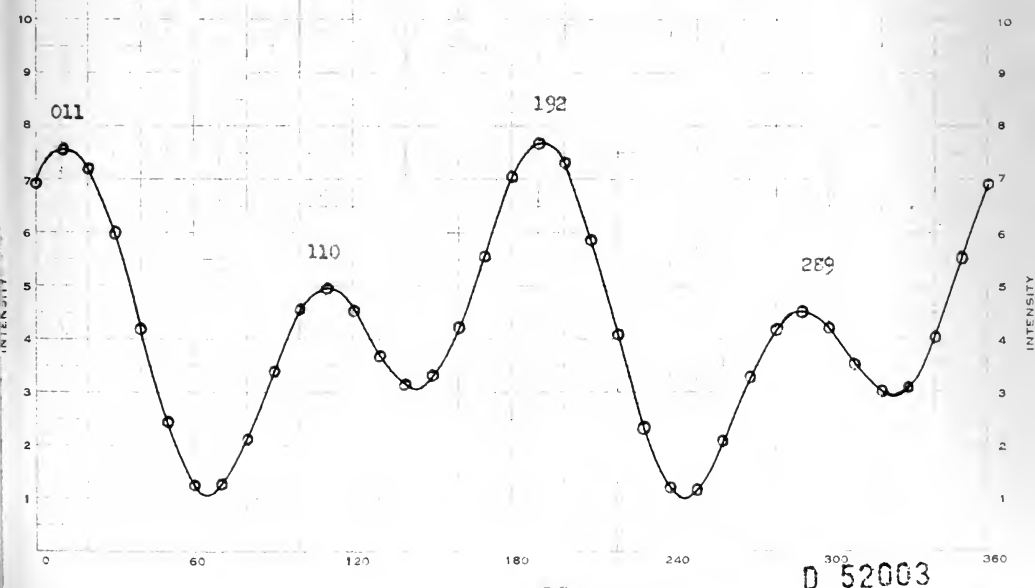
M_3	44.95	R_3	48.05
M_4	42.65	R_4	42.95
ΔM_{34}	2.30	ΔR_{34}	5.10

Av ΔM 2.60 PHASE SHIFT

Av ΔR 5.15 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1A 6 PPM N_2 DATE 30 MARCH 1952 PM 5

CRYSTAL No. 2 SURFACE FILM CONDITION 10th AUTOCLAVE OF 15 MIN. @ 233°C

@ 029		@ 122		@ 216		@ 303	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
41.8	48.0	42.9	42.6	44.0	48.4	41.9	42.6
45.0	48.4	42.3	42.8	43.6	48.3	42.3	42.5
Av. 44.90	48.20	42.60	42.70	43.80	48.35	42.10	42.55

M_1 44.90	R_1 48.20
M_2 42.60	R_2 42.70
ΔM_{12} 2.30	ΔR_{12} 5.50

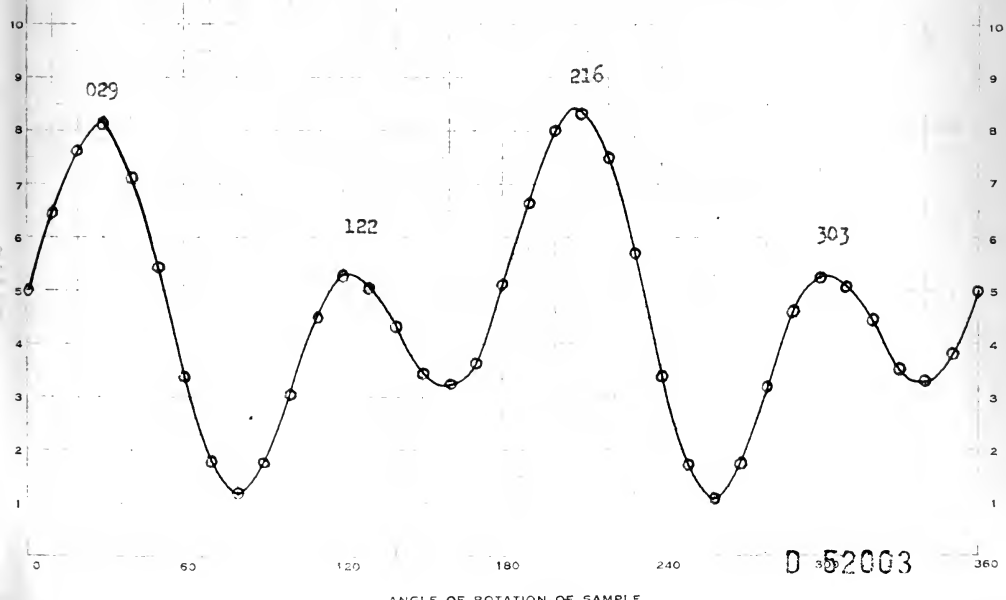
M_3 43.80	R_3 48.35
M_4 42.10	R_4 42.55
ΔM_{34} 1.70	ΔR_{34} 5.80

Av ΔM 2.00 PHASE SHIFT

Av ΔR 5.65 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1A 6 PPM N_2 DATE 1 APRIL 1952 PM

CRYSTAL No 2 SURFACE FILM CONDITION 11th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 042		@ 137		@ 222		@ 316	
	M_1	R_1	M_2	R_2	M_3	R_1	M_4	R_4
	43.0	47.8	40.9	42.8	42.7	47.9	40.7	43.0
	43.2	47.8	41.2	43.0	42.3	47.8	40.7	43.0
Av.	43.10	47.80	41.05	42.90	42.50	47.85	40.70	43.00

M_1	43.10	R_1	47.80
M_2	41.05	R_2	42.90
ΔM_{12}	2.05	ΔR_{12}	4.90

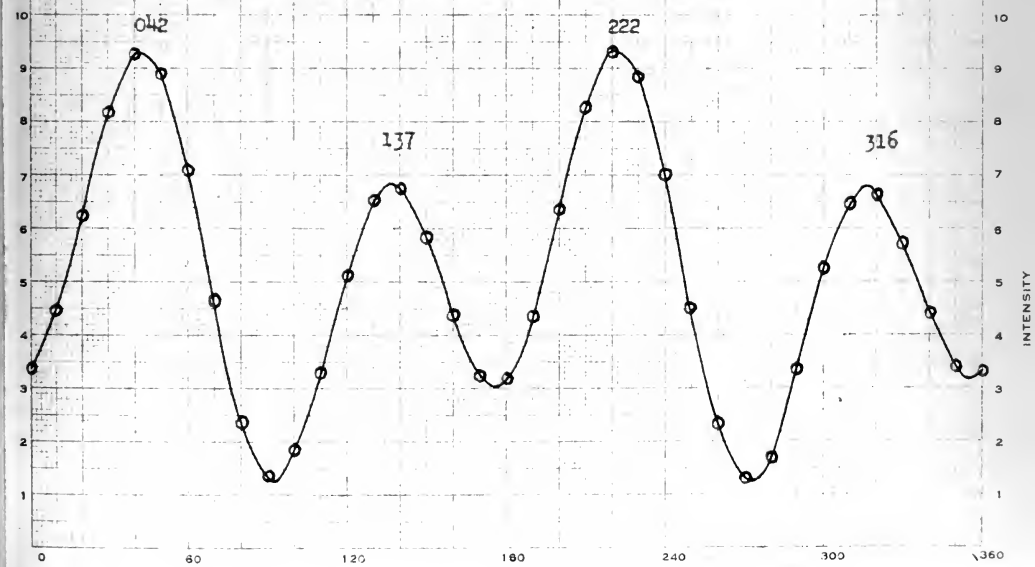
M_3	42.50	R_3	47.85
M_4	40.70	R_4	43.00
ΔM_{34}	1.80	ΔR_{34}	4.85

Av ΔM 1.93 PHASE SHIFT

Av ΔR 4.88 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1A 6 PPM Fe DATE 2 APRIL 195[illegible]

CRYSTAL No. 2 SURFACE FILM CONDITION 10th AUTOCLAVE OF 15 MIN. @ 233°C

@ 283		@ 018		@ 102		@ 198	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
43.4	48.0	41.5	43.0	42.8	47.8	41.1	43.0
43.3	47.9	41.4	42.9	43.1	48.2	41.0	43.0
Av. 43.35	47.95	41.45	42.95	42.95	48.00	41.05	43.00

M_1	43.35	R_1	47.95
M_2	41.45	R_2	42.95
ΔM_{12}	1.90	ΔR_{12}	5.00

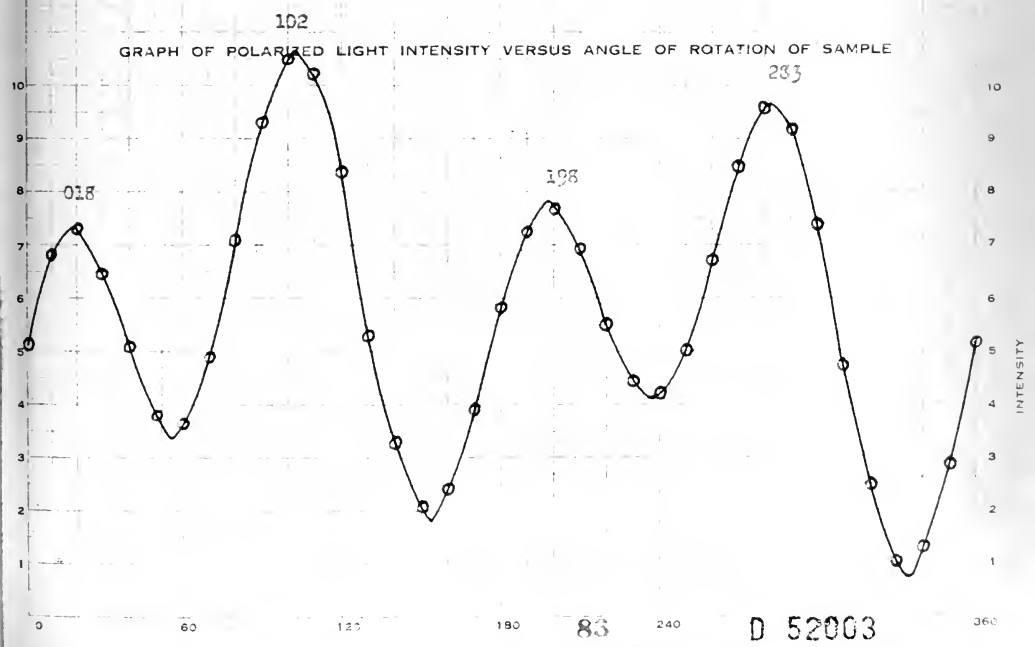
M_3	42.95	R_3	48.00
M_4	41.05	R_4	43.00
ΔM_{34}	1.90	ΔR_{34}	5.00

Av ΔM 1.90 PHASE SHIFT

Av ΔR 5.00 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1A 6 PPM N_2 DATE 3 APRIL 1952 IM

CRYSTAL No. 2 SURFACE FILM CONDITION 13th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 045		@ 138		@ 225		@ 318	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	43.1	48.1	41.1	42.7	42.7	48.0	40.8	42.8
	43.1	47.8	40.8	43.0	42.3	48.2	40.8	42.7
Av.	43.10	47.95	40.95	42.85	42.50	48.10	40.80	42.75

M_1	43.10	R_1	47.95
M_2	40.95	R_2	42.85
ΔM_{12}	2.15	ΔR_{12}	5.10

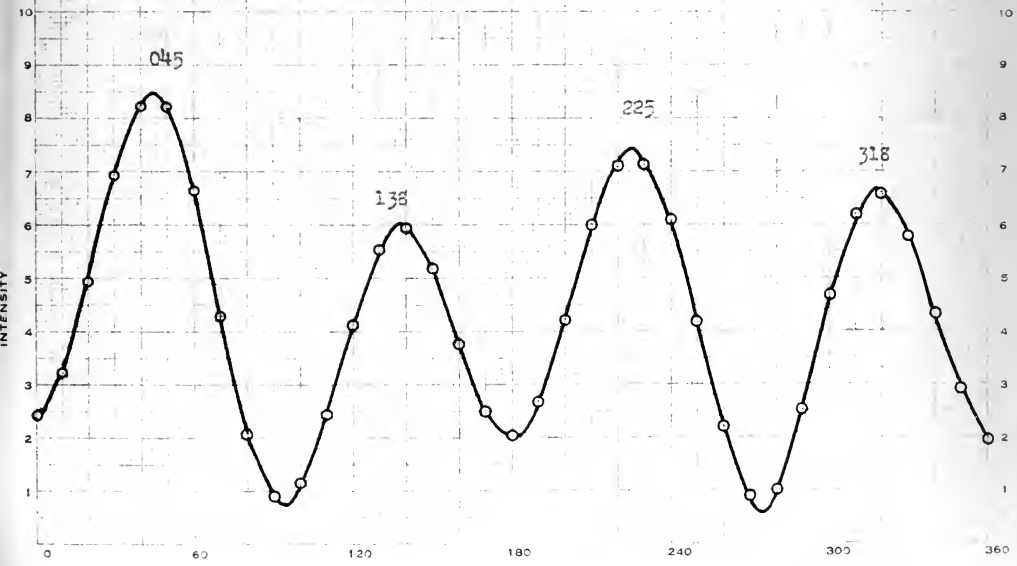
M_3	42.50	R_3	48.10
M_4	40.80	R_4	42.75
ΔM_{34}	1.70	ΔR_{34}	5.35

Av ΔM 1.93 PHASE SHIFT

Av ΔR 5.23 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 1A 6 PPM N_2 DATE 5 APRIL 1952 PM

CRYSTAL No. 2 SURFACE FILM CONDITION 15th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 046		@ 140		@ 226		@ 318	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	45.2	48.5	43.7	43.2	44.5	48.7	42.8	42.8
	44.7	48.6	43.4	43.2	44.4	48.6	42.5	43.0
Av.	45.45	48.55	43.55	43.20	44.45	48.65	42.65	42.90

M_1	45.45	R_1	48.55
M_2	43.55	R_2	43.20
ΔM_{12}	1.90	ΔR_{12}	5.35

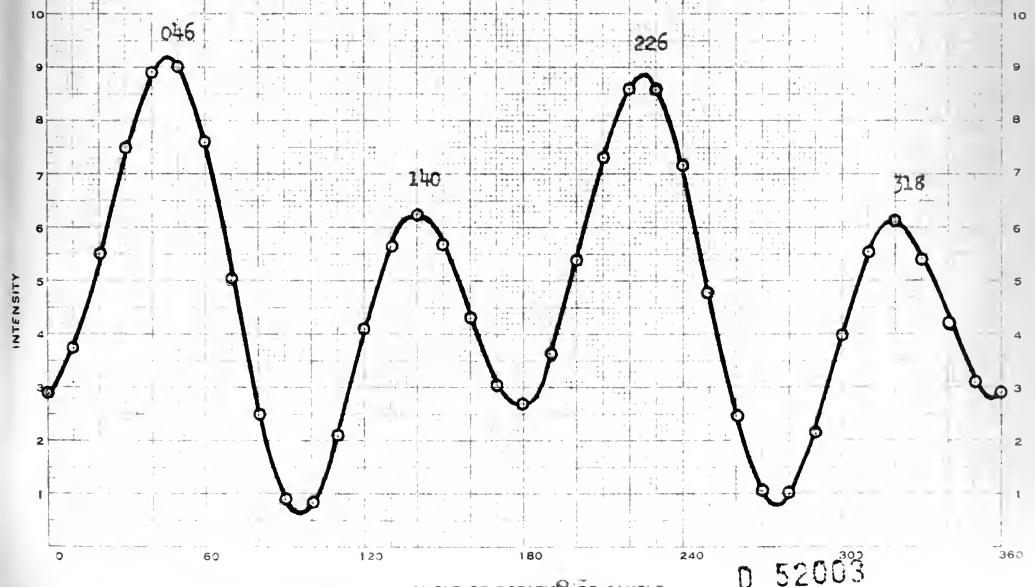
M_3	44.45	R_3	48.65
M_4	42.65	R_4	42.90
ΔM_{34}	1.80	ΔR_{34}	5.75

Av ΔM 1.85 PHASE SHIFT.

Av ΔR 5.55 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

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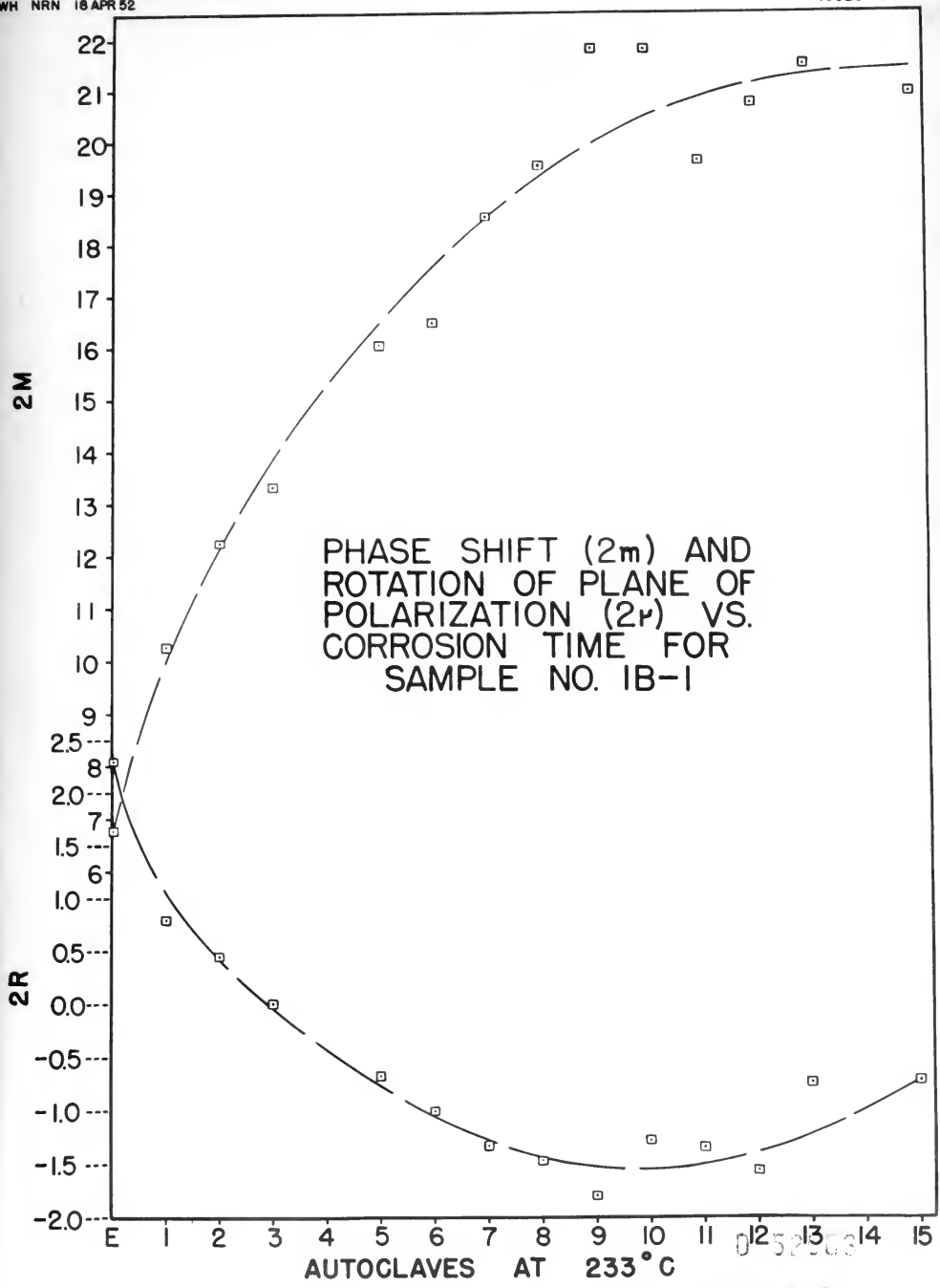


D 52503

D 52503

SAMPLE No. 1B 70 PPM N_2 DATE 5 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION AS ELECTROPOLISHED

	@ 332°		@ 242°		@ 152°		@ 062°	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	46.2	46.9	39.5	44.9	45.8	46.9	39.0	44.8
	46.4	46.9	39.4	45.0	45.8	46.9	38.8	44.8
Av.	46.30	46.90	39.45	44.95	45.80	46.90	38.90	44.80

M_1	46.30	R_1	46.90
M_2	39.45	R_2	44.95
ΔM_{12}	6.85	ΔR_{12}	1.95

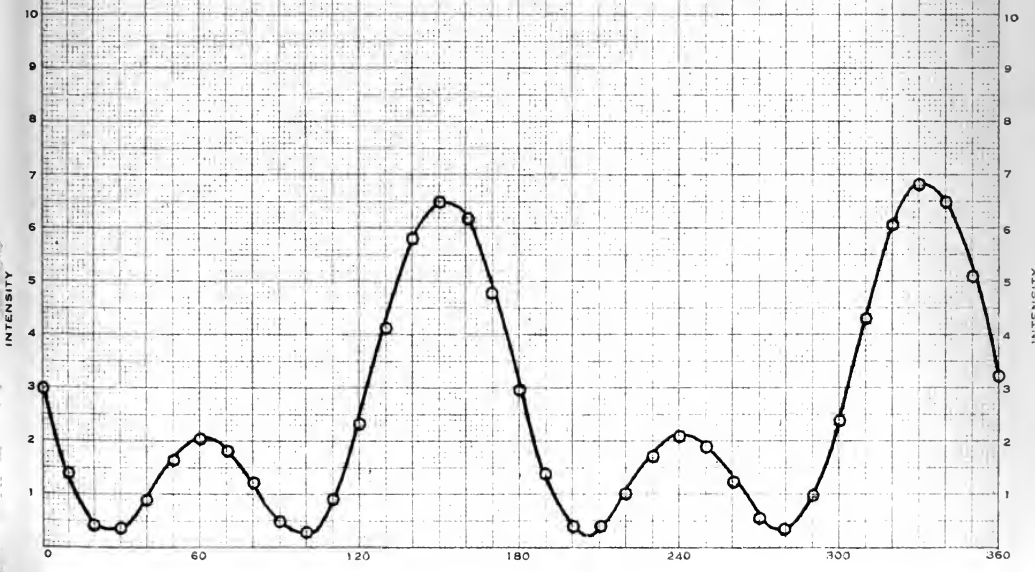
M_3	45.80	R_3	46.90
M_4	38.90	R_4	44.80
ΔM_{34}	6.70	ΔR_{31}	2.10

Av ΔM 6.77 PHASE SHIFT

Av ΔR 2.03 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1B 70 PPM N_2 DATE 13 MARCH 1952

CRYSTAL No. 1 SURFACE FILM CONDITION 1st AUTOCLAVE 15 MIN. at 233°C

@ 296		@ 206		@ 116		@ 026	
M1	R1	M2	R2	M3	R3	M4	R4
48.2	45.9	37.7	45.2	47.9	46.0	37.7	45.0
48.0	45.9	38.0	45.2	47.9	45.8	37.5	45.0
Av. 48.10	45.90	37.85	45.20	47.90	45.90	37.60	45.00

M_1	48.10	R_1	45.90
M_2	37.85	R_2	45.20
ΔM_{12}	10.25	ΔR_{12}	0.70

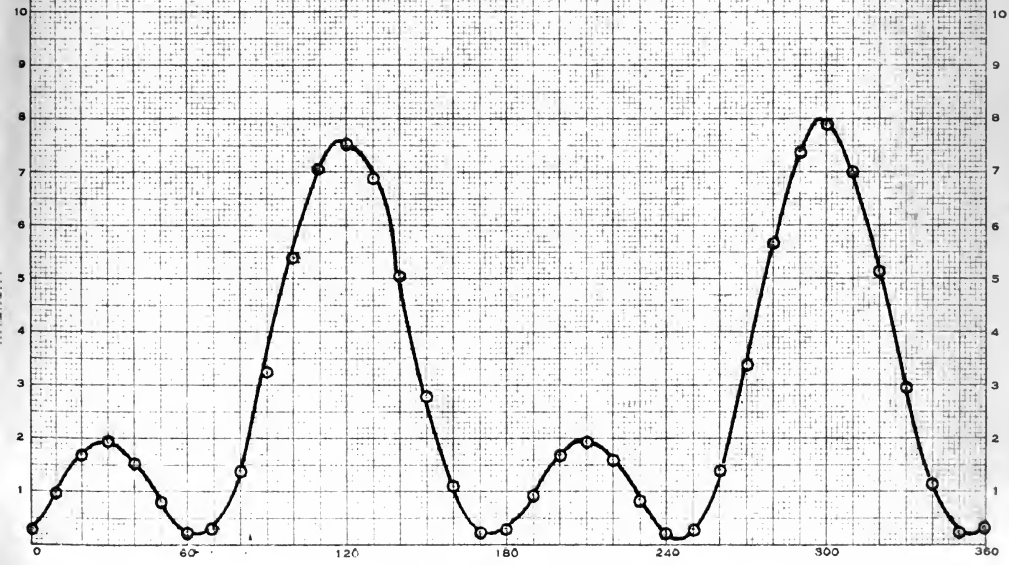
M_3	47.90	R_3	45.90
M_4	37.60	R_4	45.00
ΔM_{34}	10.30	ΔR_{34}	0.90

Av ΔM 10.27 PHASE SHIFT

Av ΔR 0.80 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

D 52003

SAMPLE No. 1B 70 PPM N_2 DATE 14 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 2nd AUTOCLAVE 15 MIN. at 233°C

	@ 275		@ 185		@ 095		@ 005	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	49.6	46.0	37.2	45.6	49.2	46.0	37.1	45.5
	49.5	46.0	37.0	45.6	49.1	46.0	37.2	45.5
Av.	49.55	46.00	37.05	45.60	49.15	46.00	37.15	45.50

M_1	49.55	R_1	46.00
M_2	37.05	R_2	45.60
ΔM_{12}	12.50	ΔR_{12}	0.40

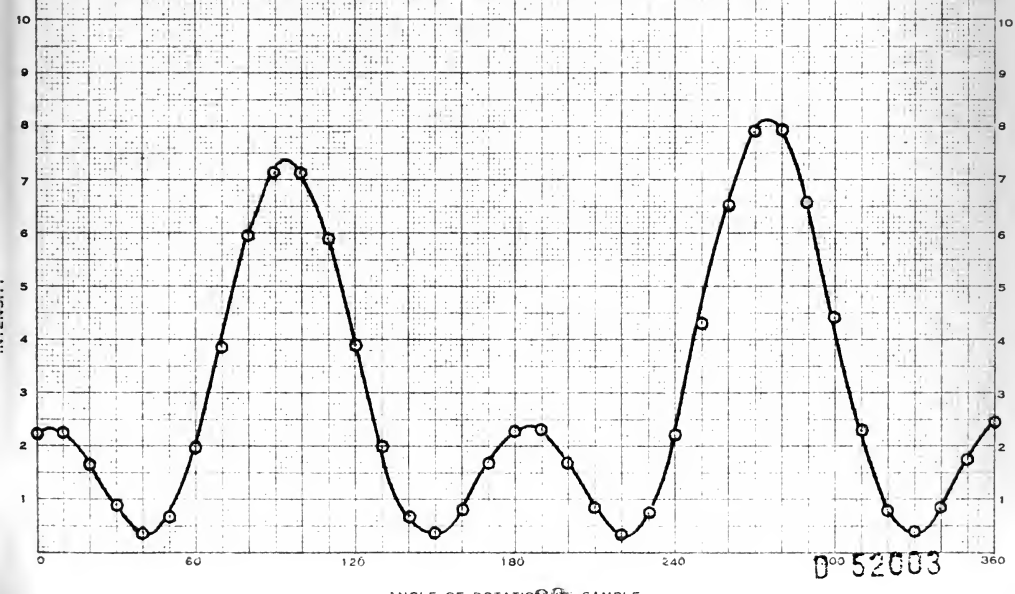
M_3	49.15	R_3	46.00
M_4	37.15	R_4	45.50
ΔM_{34}	12.00	ΔR_{34}	0.50

Av ΔM 12.25 PHASE SHIFT

Av ΔR 0.45 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1B 70 PPM N_2 DATE 17 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 3rd AUTOCLAVE 15 MIN. at 233°C

	@ 202		@ 202		@ 112		@ 022	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	50.1	45.6	36.0	46.0	49.8	46.0	35.9	45.8
	49.5	45.8	35.9	46.0	49.5	46.0	35.8	45.3
	48.9	45.8						
	48.7	45.8						
	48.8	45.8						
Av.	49.20	45.80	35.95	46.0	49.65	46.0	35.85	45.80

M_1	49.20	R_1	45.80
M_2	35.95	R_2	46.00
ΔM_{12}	13.25	ΔR_{12}	-0.20

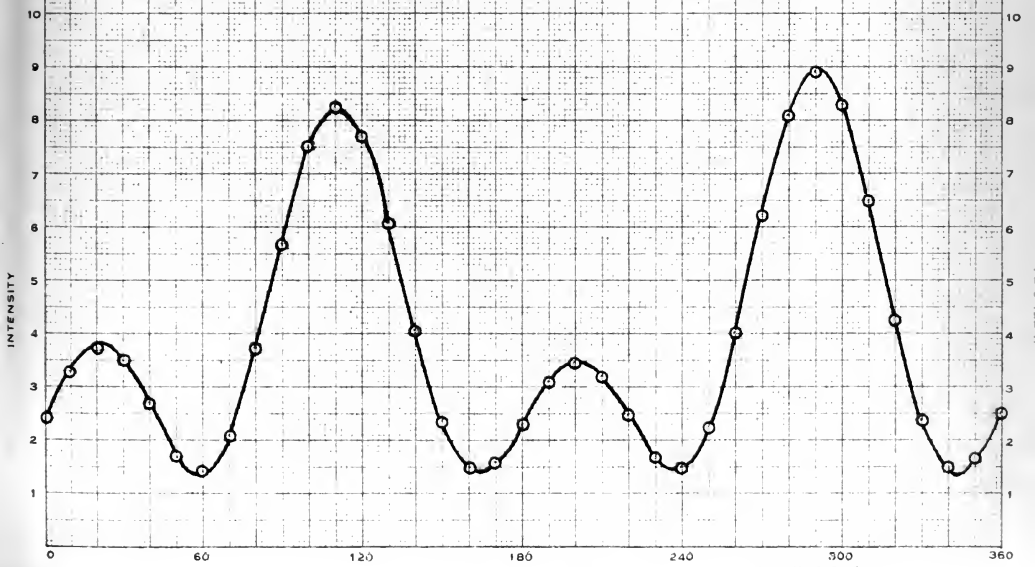
M_3	49.65	R_3	46.00
M_4	35.85	R_4	45.80
ΔM_{34}	13.40	ΔR_{34}	0.20

Av ΔM 13.32 PHASE SHIFT

Av ΔR 0.0 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1B 70 PPM H_2 DATE 20 MARCH 195? AM

CRYSTAL No. SURFACE FILM CONDITION 4th AUTOCLAVE 15 MIN. @ 233°C

	@ 282		@ 192		@ 102		@ 012	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	48.8	45.3	35.0	45.6	48.8	45.2	35.0	45.5
	48.6	45.5	34.9	45.6	48.6	45.4	34.7	45.7
Av.	48.70	45.40	34.9	45.6	48.70	45.30	34.85	45.60

M_1 48.70	R_1 45.40
M_2 34.90	R_2 45.60
ΔM_{12} 13.80	ΔR_{12} −0.20

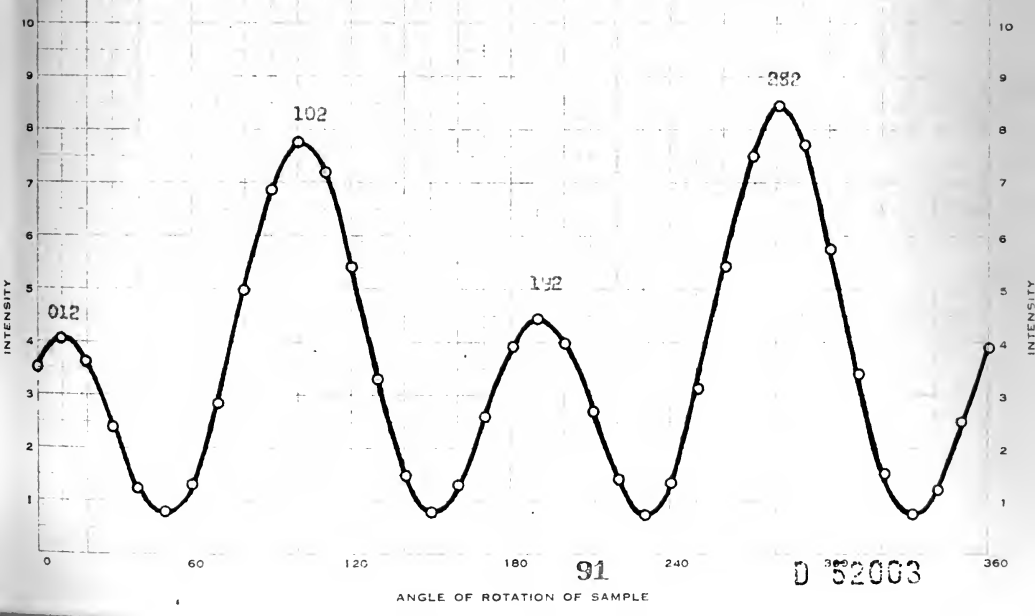
M_3 48.70	R_3 45.30
M_4 34.85	R_4 45.60
ΔM_{34} 13.85	ΔR_{34} −0.30

Av ΔM 13.82 PHASE SHIFT

Av ΔR −0.25 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 13 10 PPM N_2 DATE 24 MARCH 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 5th AUTOCLAVE 15 MIN. @ 233°C

@ 254		@ 164		@ 074		@ 344	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
49.9	45.2	33.6	45.8	49.3	45.1	33.5	45.8
49.6	45.2	33.5	45.9	49.6	45.2	33.6	45.8
Av. 49.75	45.20	33.55	45.85	49.45	45.10	33.55	45.80

M_1 49.75 R_1 45.20
M_2 33.55 R_2 45.85
ΔM_{12} 16.20 ΔR_{12} -0.65

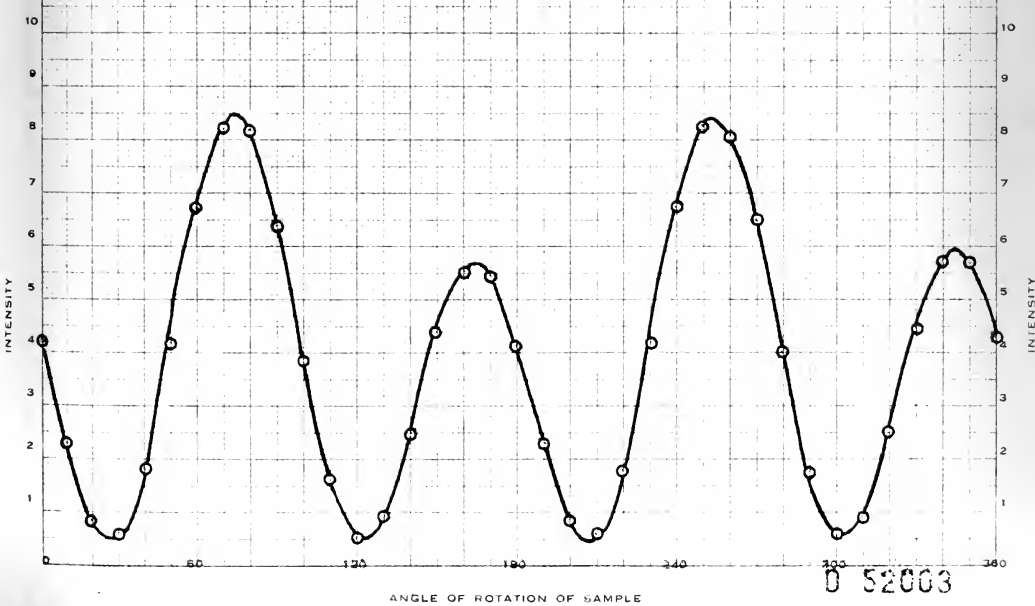
M_3 49.45 R_3 45.10
M_4 33.55 R_4 45.80
ΔM_{34} 15.90 ΔR_{34} -0.70

Av ΔM 16.05 PHASE SHIFT

Av ΔR -0.68 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 13 70 PPM N_2 DATE 25 MARCH 1952 PM

CRYSTAL No 1 SURFACE FILM CONDITION 6th AUTOCLAVE 15 MIN. @ 233°C

	@ 356		@ 256		@ 176		@ 086	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	51.3	45.2	35.0	46.0	51.5	45.0	35.4	46.0
	51.5	45.0	34.8	46.0	51.3	44.8	34.9	46.1
							34.7	46.0
Av.	51.40	45.10	34.90	46.00	51.40	44.90	35.00	46.03

M_1	51.40	R_1	45.10
M_2	34.90	R_2	46.00
ΔM_{12}	16.50	ΔR_{12}	-0.90

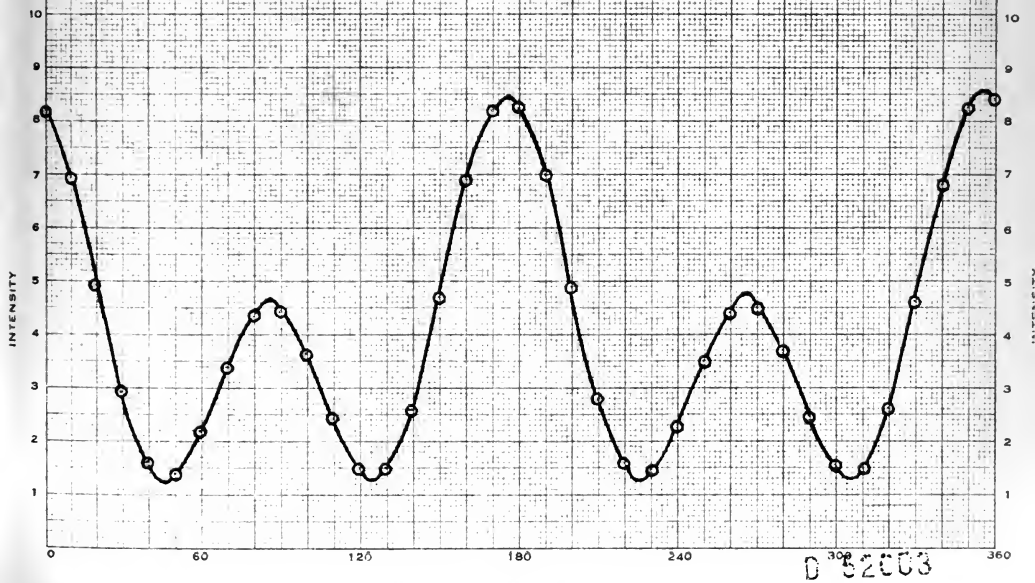
M_3	51.40	R_3	44.90
M_4	35.00	R_4	46.03
ΔM_{34}	16.40	ΔR_{34}	-1.13

Av ΔM 16.45 PHASE SHIFT

Av ΔR -1.01 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 13 70 PPM N_2 DATE 26 MARCH 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 7th AUTOCLAVE 15 MIN. @ 233°C

	@ 306		@ 216		@ 126		@ 036	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	52.8	44.9	34.2	46.1	52.2	44.8	33.9	46.2
	52.7	44.7	34.0	46.2	52.1	44.8	33.7	46.1
Av.	52.75	44.80	34.10	46.15	52.15	44.80	33.80	46.10

M_1	52.75	R_1	44.80
M_2	34.10	R_2	46.15
ΔM_{12}	18.65	ΔR_{12}	-1.35

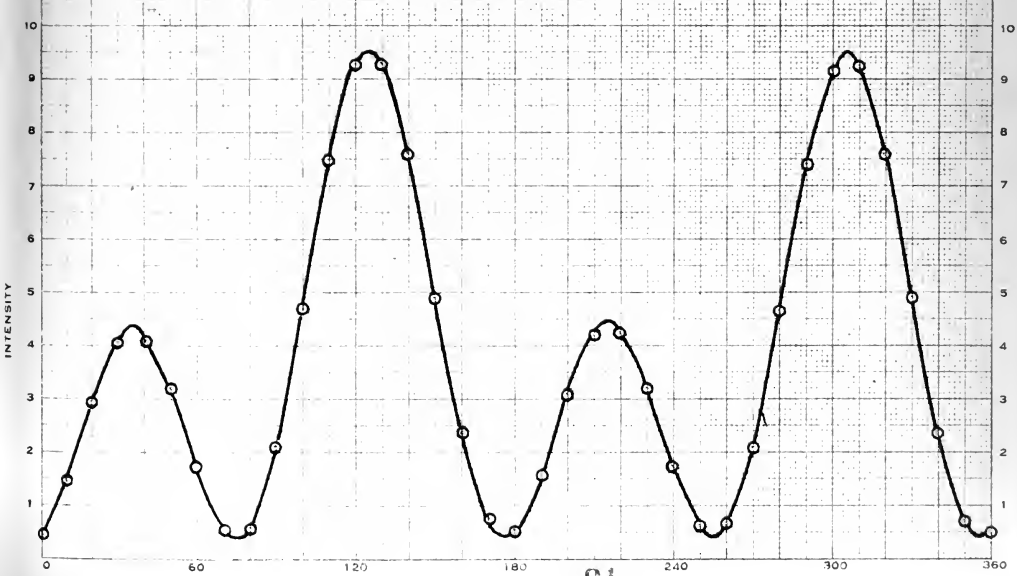
M_3	52.15	R_3	44.80
M_4	33.80	R_4	46.10
ΔM_{34}	18.35	ΔR_{34}	-1.30

Av ΔM 18.50 PHASE SHIFT

Av ΔR -1.33 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1B 70 PPM N_2 DATE 27 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 8th AUTOCLAVE 15 MIN. @ 233°C

	@ 328		@ 238		@ 148		@ 058	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	53.2	44.7	33.6	46.1	52.7	44.8	33.2	46.2
	53.1	44.8	33.4	46.3	52.7	44.7	33.5	46.3
Av.	53.15	44.75	33.50	46.20	52.70	44.75	33.35	46.25

M_1	53.15	R_1	44.75
M_2	33.50	R_2	46.20
ΔM_{12}	19.65	ΔR_{12}	-1.45

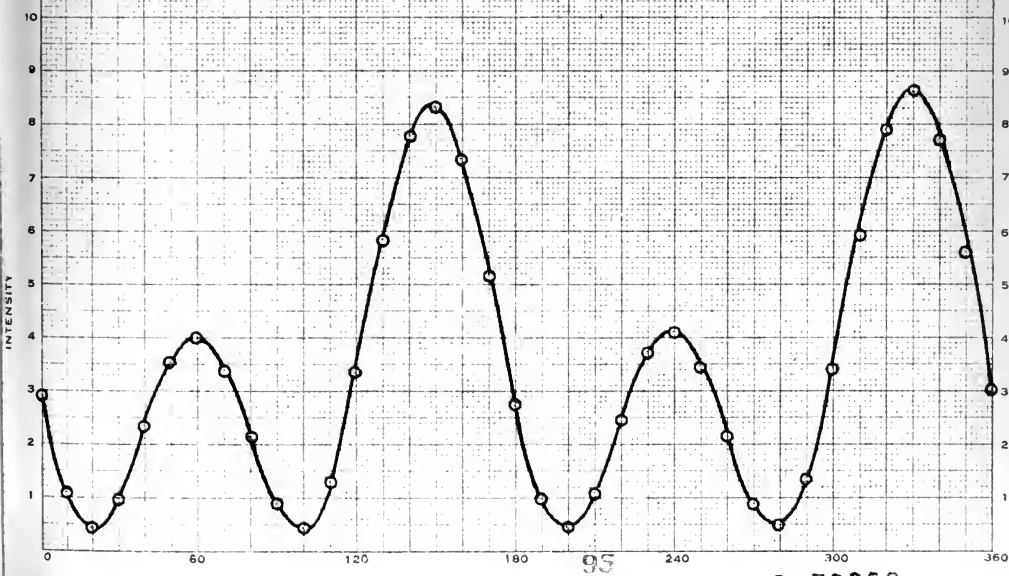
M_3	52.70	R_3	44.75
M_4	33.35	R_4	46.25
ΔM_{34}	19.35	ΔR_{34}	-1.50

Av ΔM 19.50 PHASE SHIFT

Av ΔR -1.47 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1B 70 PPM N_2 DATE 28 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 9th AUTOCLAVE 15 MIN. @ 233°C

	@ 205		@ 115		@ 025		@ 295	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	56.2	44.4	34.2	46.6	55.5	45.0	34.3	46.5
	56.5	44.6	34.2	46.5	55.8	44.8	34.1	46.6
Av.	56.35.	44.50	34.20	46.55	55.65	44.90	34.20	46.55

M_1	56.35	R_1	44.50
M_2	34.20	R_2	46.55
ΔM_{12}	22.15	ΔR_{12}	-2.05

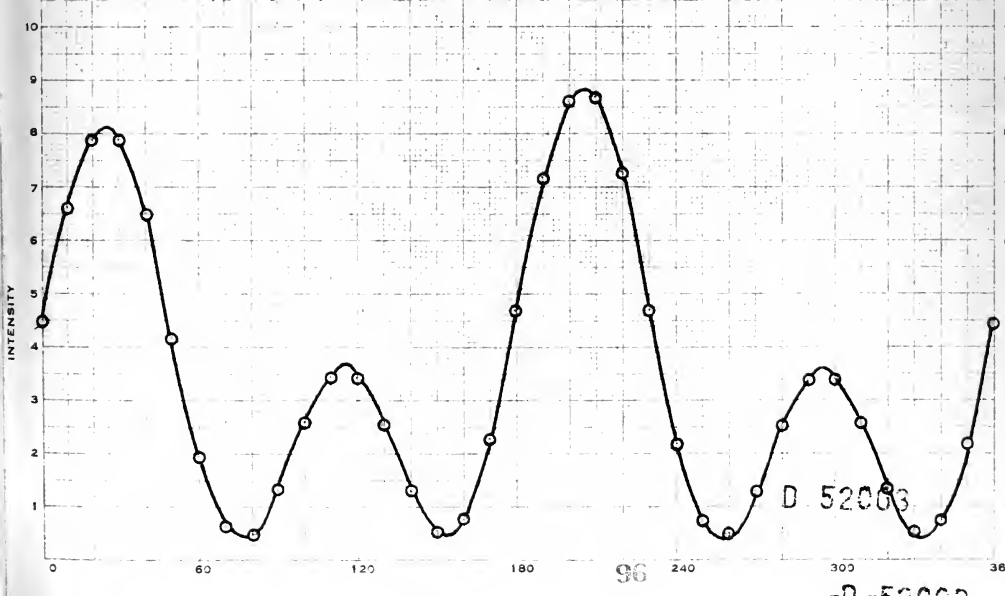
M_3	55.65	R_3	44.90
M_4	34.20	R_4	46.55
ΔM_{34}	21.45	ΔR_{31}	-1.65

Av ΔM 21.80 PHASE SHIFT

Av ΔR -1.80 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52063

SAMPLE No 1B 70 PPM N_2 DATE 31 MARCH 1952 AM

CRYSTAL No 1 SURFACE FILM CONDITION 10th AUTOCLAVE 15 MIN. @ 233°C

	@ 304		@ 214		@ 124		@ 034	
	M1	R1	M2	R2	M3	R3	M4	R4
	55.6	44.9	34.0	46.2	55.7	45.0	33.6	46.4
	55.5	45.1	34.0	46.3	55.8	45.1	33.9	46.3
Av.	55.55	45.00	34.00	46.25	55.75	45.05	33.75	46.35

M_1	55.55	R_1	45.00
M_2	34.00	R_2	46.25
ΔM_{12}	21.55	ΔR_{12}	-1.25

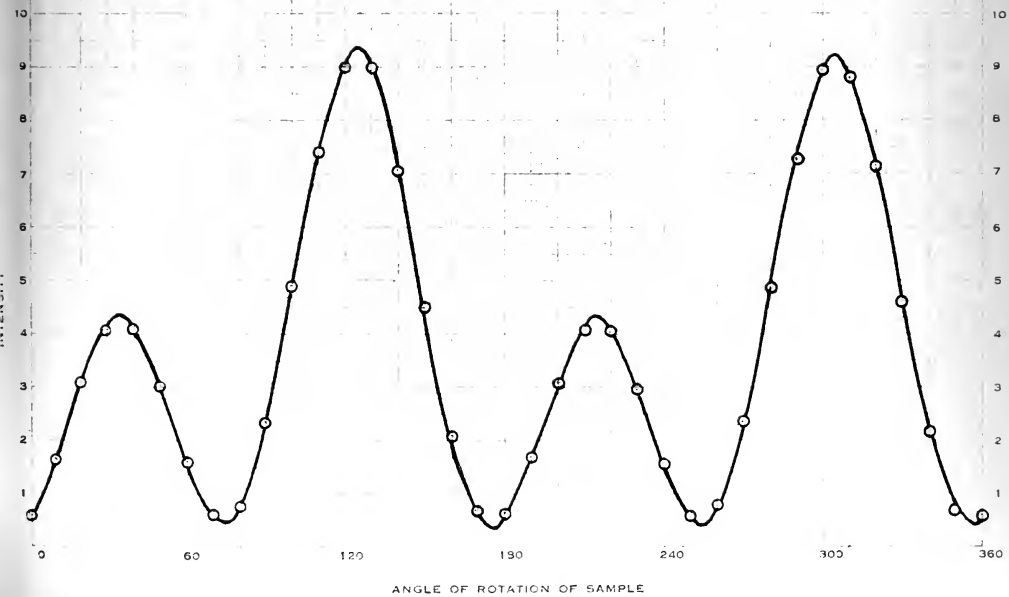
M_3	55.75	R_3	45.05
M_4	33.75	R_4	46.35
ΔM_{34}	22.00	ΔR_{34}	-1.30

Av ΔM 21.77 PHASE SHIFT

Av ΔR -1.28 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



SAMPLE No 13 90 PPM N_2 DATE 1 APRIL 1952 AM

CRYSTAL No 1 SURFACE FILM CONDITION 11th AUTOCLAVE 15 MIN. @ 233°C

@ 333		@ 243		@ 153		@ 063	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
52.4	45.0	33.2	46.6	52.4	45.1	32.4	46.2
52.3	44.9	32.9	46.5	52.6	45.2	32.8	46.3
Av. 52.35	44.95	33.05	46.55	52.50	45.15	32.60	46.25

M_1	52.35	R_1	44.95
M_2	33.05	R_2	46.55
ΔM_{12}	19.30	ΔR_{12}	-1.60

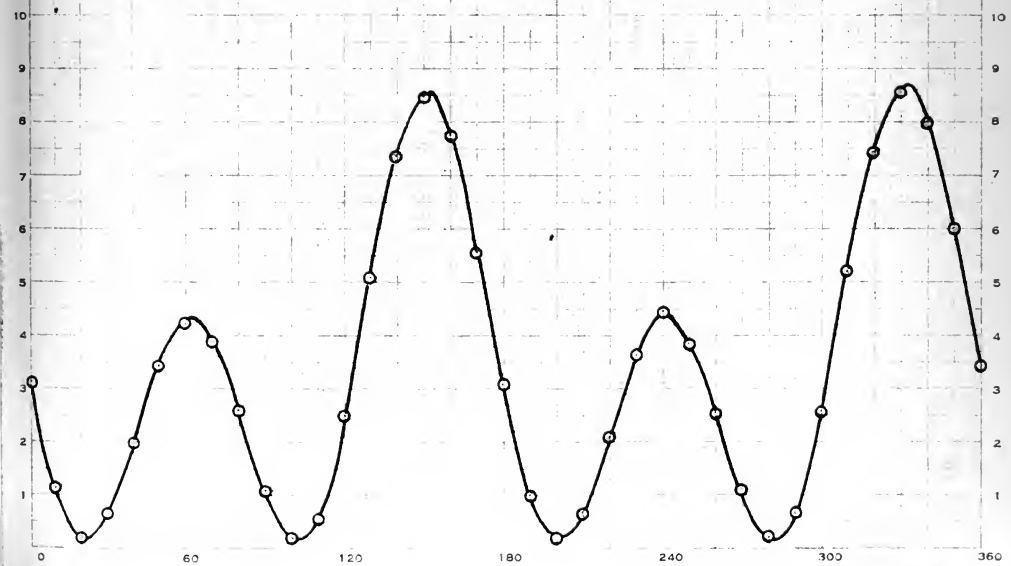
M_3	52.50	R_3	45.15
M_4	32.60	R_4	46.25
ΔM_{34}	19.90	ΔR_{34}	-1.10

Av ΔM 19.60 PHASE SHIFT

Av ΔR -1.35 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003 D 52003

SAMPLE No. 1B 90 PPM N_2 DATE 4 APRIL 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 13th AUTOCLAVE 15 MIN. @ 233°C

@ 214		@ 124		@ 034		@ 304	
M1	R1	M2	R2	M3	R3	M4	R4
52.6	45.4	30.9	46.1	52.3	45.3		
52.6	45.6	31.2	46.4				
V. 52.60	45.50	31.15	46.25				

M_1 52.60 R_1 45.50

M_2 31.15 R_2 46.25

ΔM_{12} 21.45 ΔR_{12} - .75

M_3 R_3

M_4 R_4

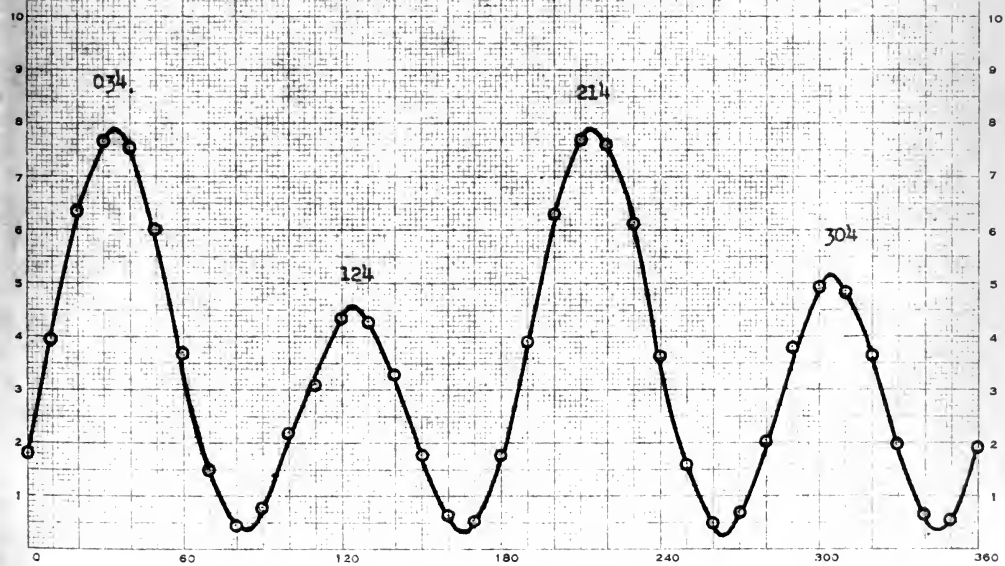
ΔM_{34} ΔR_{32}

Av ΔM PHASE SHIFT

Av ΔR PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 15 90 PPM N_2 DATE 2 APRIL 1952 AM

CRYSTAL No 1 SURFACE FILM CONDITION 12th AUTOCLAVE 15 MIN. @ 233°C

	@ 253		@ 163		@ 073		@ 343	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	53.6	44.9	32.5	46.6	53.4	44.8	32.9	46.5
	53.5	45.1	32.8	46.4	53.2	45.0	32.7	46.6
Av.	53.55	45.00	32.65	46.50	53.30	44.90	32.80	46.55

M	53.55	R	45.00
M_2	32.65	R_2	46.50
ΔM_{12}	20.50	ΔR_{12}	-1.65

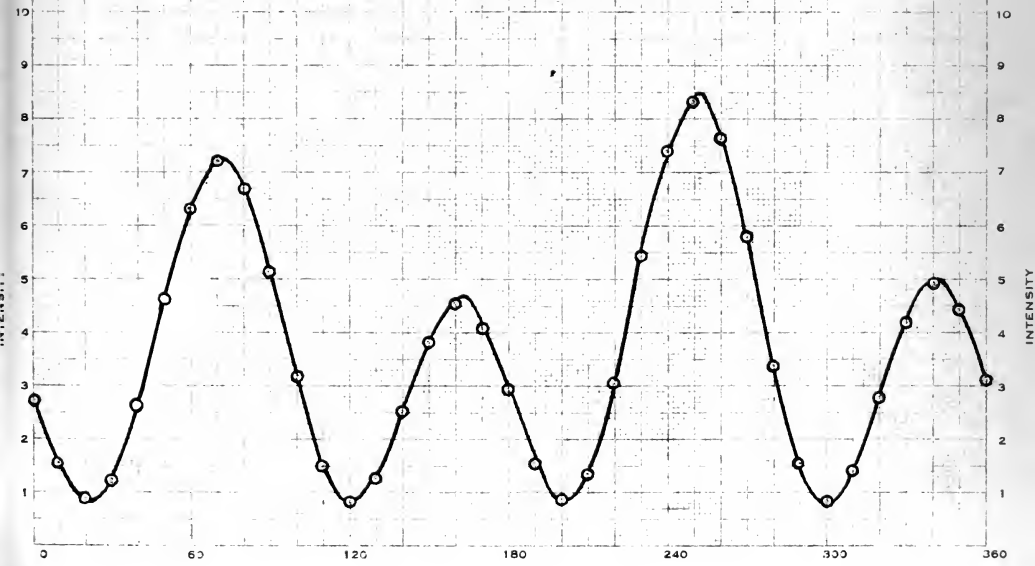
M_3	53.30	R_3	44.90
M_4	32.80	R_4	46.55
ΔM_{34}	20.50	ΔR_{34}	-1.65

Av ΔM 20.70 PHASE SHIFT

Av ΔR -1.57 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1B 70 PPM N_2 DATE 5 APRIL 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 15th AUTOCLAVE OF 15 MIN. @ 233°C

@ 334		@ 244		@ 154		@ 064	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
55.7	45.1	34.3	45.7	54.3	45.3	34.2	46.0
55.6	44.9	34.2	45.9	54.7	45.2	34.0	45.8
Av. 55.65	45.00	34.25	45.80	54.50	45.25	34.10	45.90

M_1 55.65 R_1 45.00
M_2 34.25 R_2 45.80
ΔM_{12} 21.40 ΔR_{12} = .80

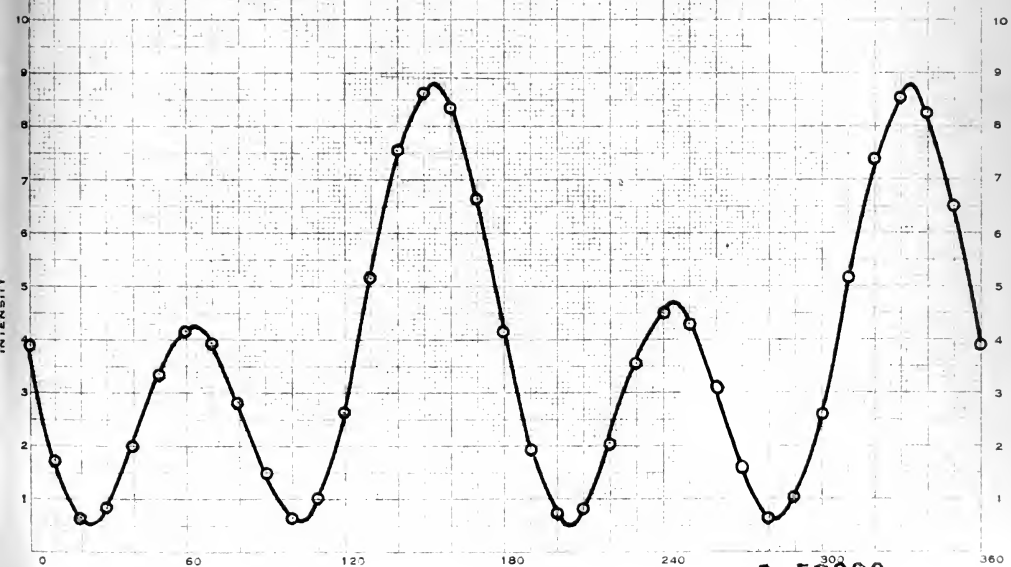
M_3 54.50 R_3 45.25
M_4 34.10 R_4 45.90
ΔM_{34} 20.40 ΔR_{31} = .65

Av ΔM 20.90 PHASE SHIFT

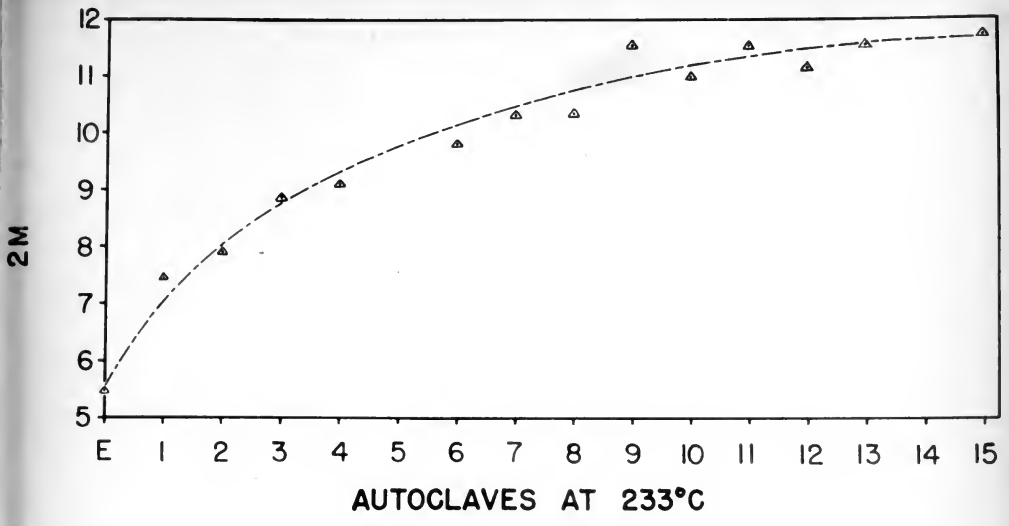
Av ΔR -0.72 PHASE ROTATION

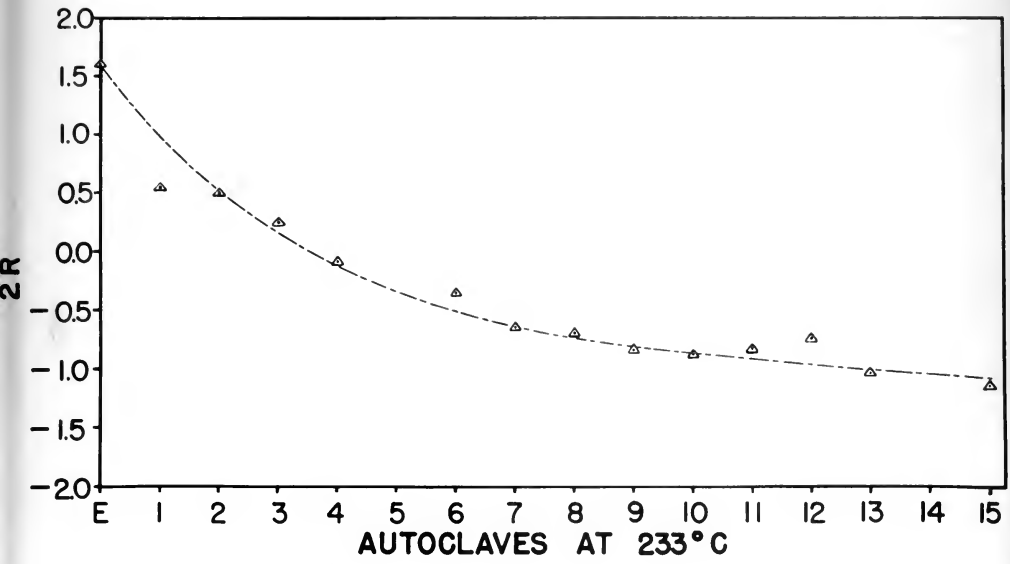
PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





PHASE SHIFT (2M) AND ROTATION OF PLANE OF POLARIZATION (2R) VS. CORROSION TIME FOR SAMPLE NO. 1C-1

SAMPLE No. 1C 113 PPM N_2 DATE 5 March 1962 PM

CRYSTAL No 1 SURFACE FILM CONDITION AS ELECTROPOLISHED

	@ 306		@ 036		@ 126		@ 216	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	44.5	46.6	39.2	45.0	45.5	46.7	39.8	44.9
	44.8	46.7	39.6	45.2	45.3	46.7	39.6	45.2
Av.	44.65	46.65	39.40	45.10	45.40	46.70	39.70	45.05

M_1	44.65	R_1	46.65
M_2	39.40	R_2	45.10
ΔM_{12}	5.25	ΔR_{12}	1.55

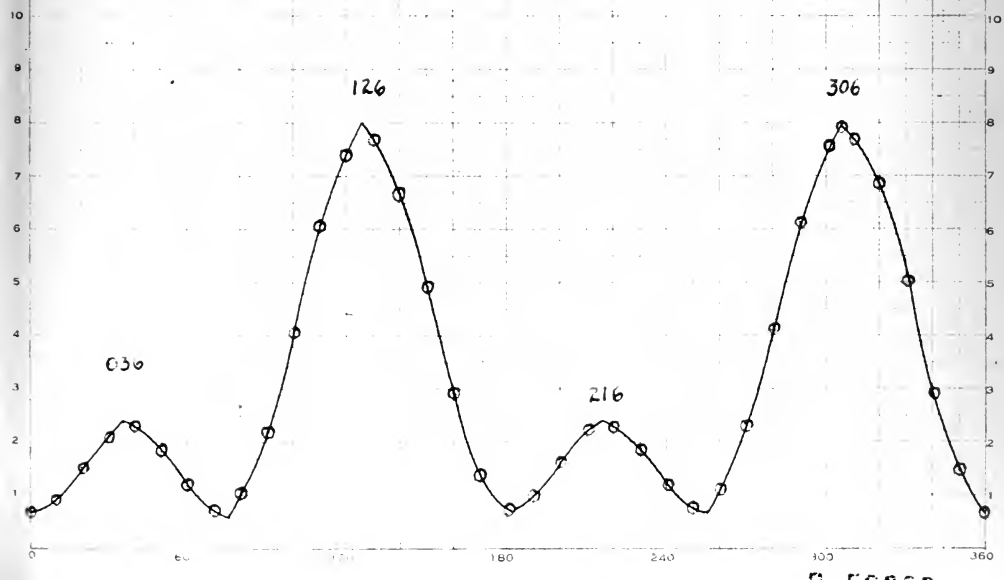
M_3	45.40	R_3	46.70
M_4	39.70	R_4	45.05
ΔM_{34}	5.70	ΔR_{31}	1.65

Av ΔM 5.48 PHASE SHIFT

Av ΔR 1.60 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 1C 113 PPM N_2 DATE 12 March 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 1st AUTOCLAVE 15 Min at 233°C

@ 306		@ 036		@ 126		@ 216	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
45.5	45.9	38.0	45.1	45.8	45.8	38.7	45.0
45.4	45.7	38.0	45.4	45.7	45.5	37.9	45.2
AV. 45.45	45.80	38.00	45.25	45.75	45.65	38.30	45.10

M_1	45.45	R_1	45.80
M_2	38.00	R_2	45.25
ΔM_{12}	7.45	ΔR_{12}	0.55

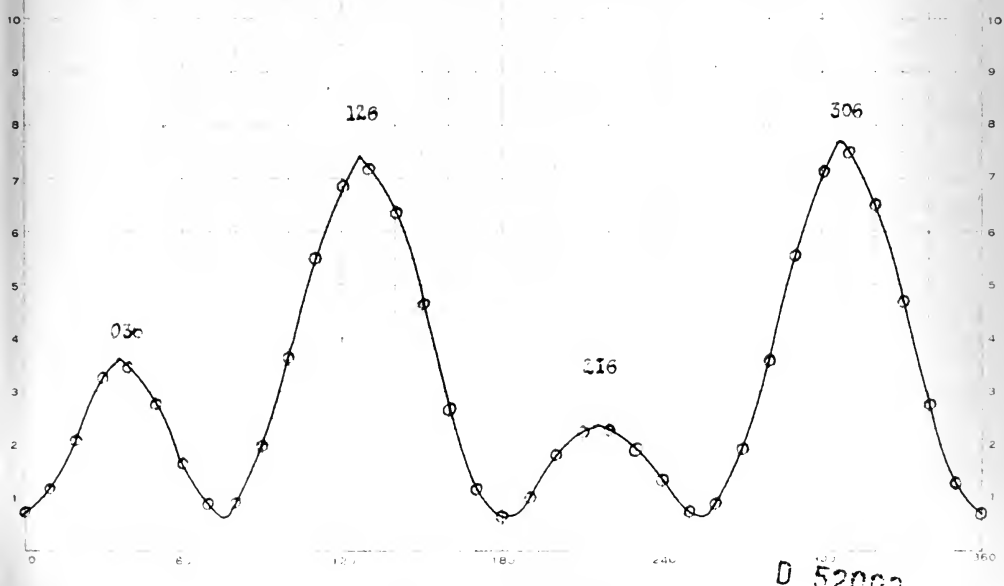
M_3	45.75	R_3	45.65
M_4	38.30	R_4	45.10
ΔM_{34}	7.45	ΔR_{34}	0.55

Av ΔM 7.45 PHASE SHIFT

Av ΔR 0.55 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 10 113 PPM No DATE 14 MARCH 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 2nd AUTOCLAVE OF 15 Min. at 233°C

@ 060		@ 150		@ 240		@ 330	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
46.0	46.2	37.9	45.7	46.2	46.1	39.3	45.6
45.0	46.0	38.0	45.5	46.3	46.3	38.7	45.8
AV. 46.00	46.10	37.95	45.60	46.25	46.20	39.00	45.70

M_1	46.00	R_1	46.10
M_2	37.95	R_2	45.60
ΔM_{12}	8.05	ΔR_{12}	.50

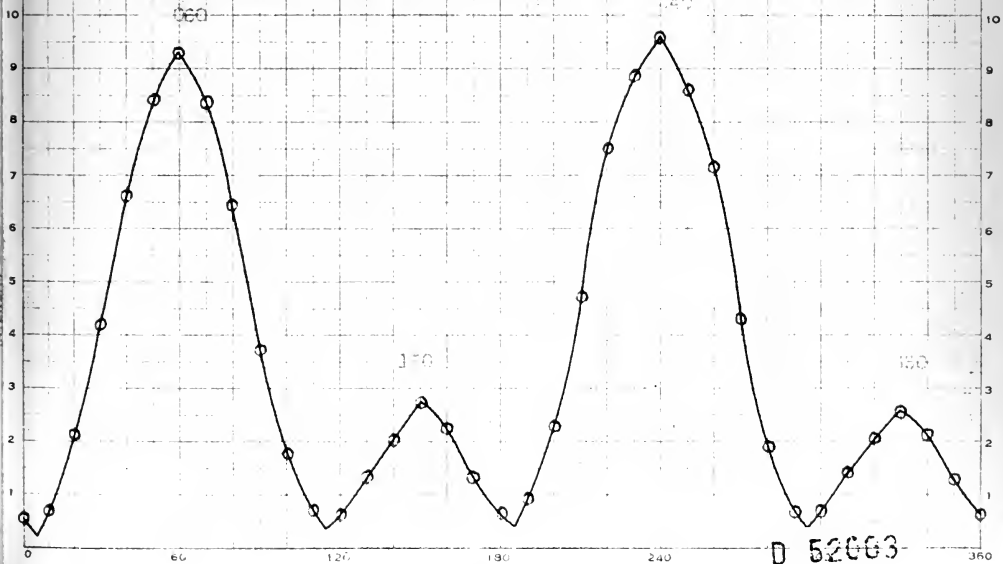
M_3	46.25	R_3	46.20
M_4	38.50	R_4	45.70
ΔM_{34}	7.75	ΔR_{34}	.50

Av ΔM 7.90 PHASE SHIFT

Av ΔR .50 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

ANGLE OF ROTATION OF SAMPLE

SAMPLE No [illegible] 115 PPM .2 DATE [illegible]

CRYSTAL No. 1 SURFACE FILM CONDITION [illegible]

	@ 315		@ 340		@ 336		@ [illegible]	
	M1	R1	M2	R2	M3	R3	M4	R4
	46.4	46.0	39.2	45.3	47.2	45.9	39.0	45.6
	47.0	46.0	39.2	45.7	47.9	46.0	39.1	45.8
AV.	46.70	45.95	39.10	45.55	47.55	45.90	39.05	45.70

M_1	46.00	R_1	45.95
M_2	39.10	R_2	45.55
ΔM_{12}	6.90	ΔR_{12}	.30

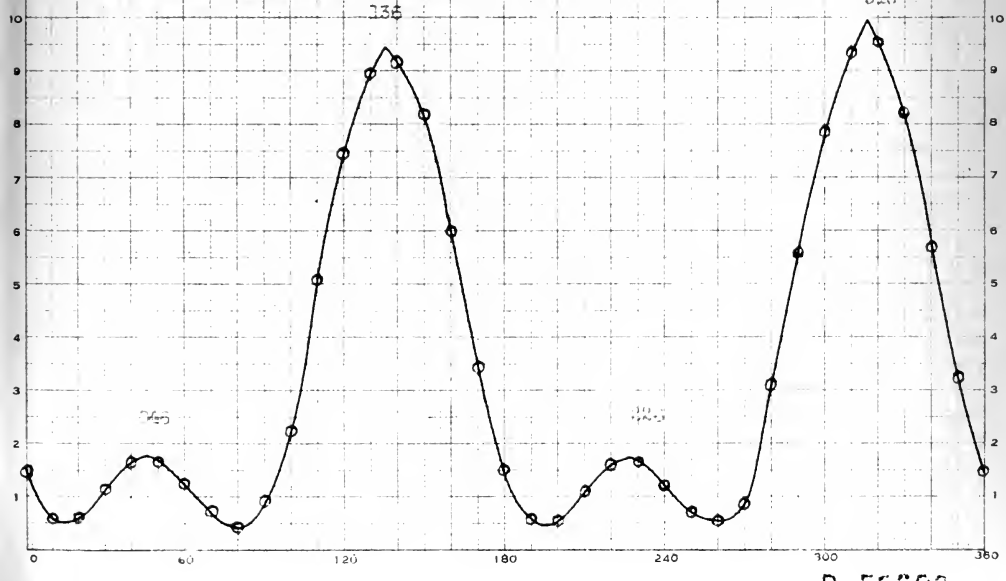
M_3	47.05	R_3	45.90
M_4	39.05	R_4	45.70
ΔM_{34}	8.00	ΔR_{34}	.20

Av ΔM 8.5 PHASE SHIFT

Av ΔR .25 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 10 113 PPM [illegible] DATE [illegible] MARCH [illegible]

CRYSTAL No 1 SURFACE FILM CONDITION 4th AUTOCLAVE OF 15 MIN. at 23[illegible]

	@ 289		@ 019		@ 109		@ 199	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	46.4	45.3	37.1	45.1	46.0	45.2	37.2	45.2
	46.2	45.1	37.2	45.5	46.0	45.1	36.8	45.2
Av.	46.30	45.20	37.15	45.30	46.00	45.15	37.00	45.20

M_1	46.30	R_1	45.20
M_2	37.15	R_2	45.30
ΔM_{12}	9.15	ΔR_{12}	- .10

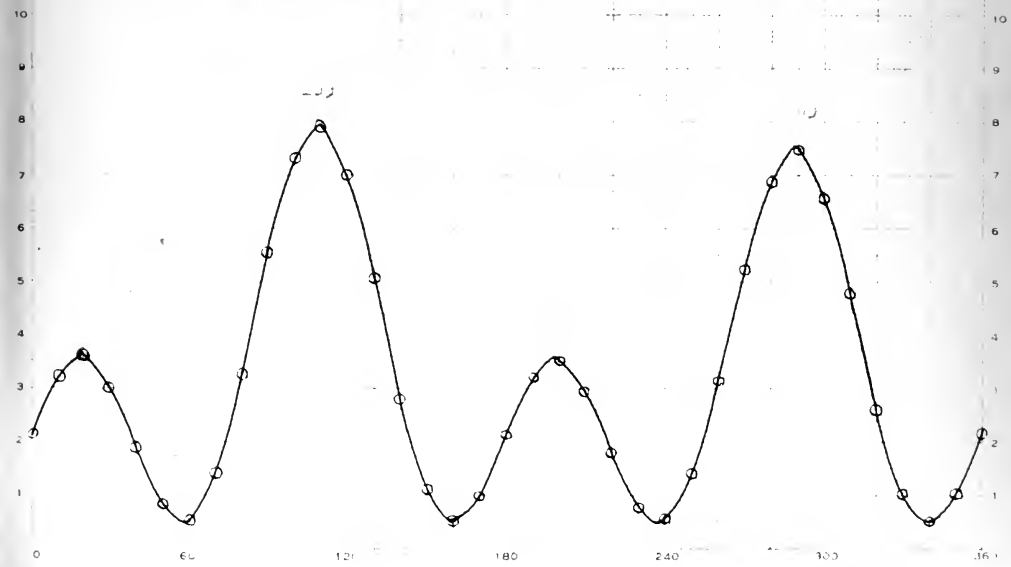
M_3	46.00	R_3	45.15
M_4	37.00	R_4	45.20
ΔM_{34}	9.00	ΔR_{34}	- .05

Av ΔM 9.08 PHASE SHIFT

Av ΔR -.08 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No [illegible] PPM [illegible] DATE 25 MARCH 1952 PM

CRYSTAL No [illegible] SURFACE FILM CONDITION [illegible] IN AUTOCLAVE OF 15 MIN. at 233°C

	@ 343		@ 073		@ 163		@ 253	
	M	R	M	R	M	R	M	R
	48.5	45.6	38.6	45.7	47.9	45.4	38.2	45.7
	48.4	45.5	38.4	46.0	47.2	45.5	38.2	46.0
Av	48.45	45.55	38.50	45.85	47.55	45.45	38.20	45.85

M_1	48.45	R_1	45.55
M_2	38.50	R_2	45.85
ΔM_{12}	9.95	ΔR_{12}	- .30

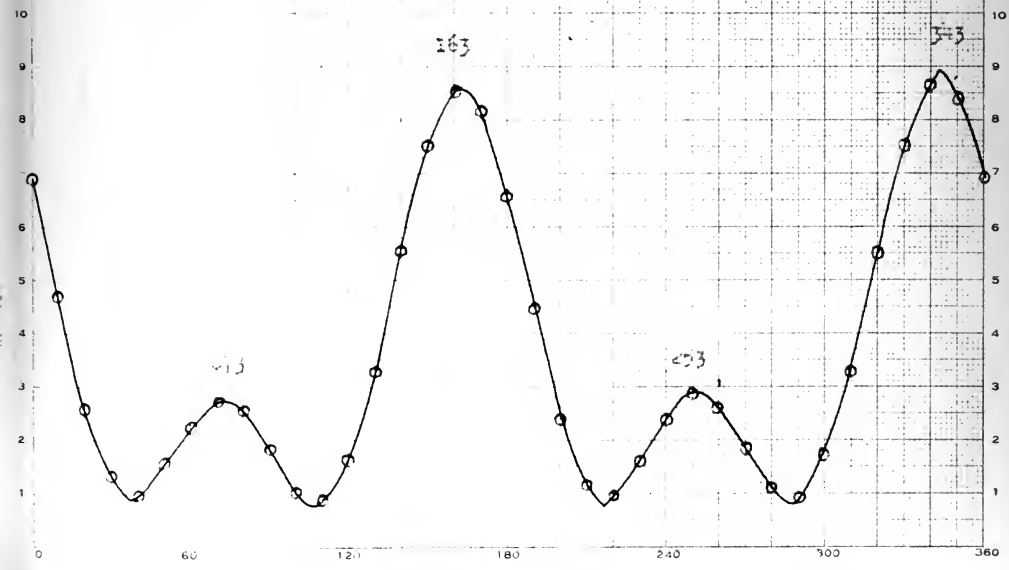
M_3	47.85	R_3	45.45
M_4	38.20	R_4	45.85
ΔM_{34}	9.65	ΔR_{34}	- .40

Av ΔM 9.80 PHASE SHIFT

Av ΔR -.35 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 13 113 PPM H_2 DATE 25 MARCH 1962 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 7th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 335		@ 065		@ 155		@ 245	
	M_1	R_1	M_4	R_2	M_3	R_3	M_4	R_4
	48.5	45.2	38.0	45.8	48.1	45.2	37.6	45.7
	48.4	45.1	38.2	45.7	48.1	45.1	38.1	46.0
Av.	48.45	45.15	38.10	45.75	48.10	45.15	37.85	45.35

M_1	48.45	R_1	45.15
M_2	38.10	R_2	45.75
ΔM_{12}	10.35	ΔR_{12}	- .60

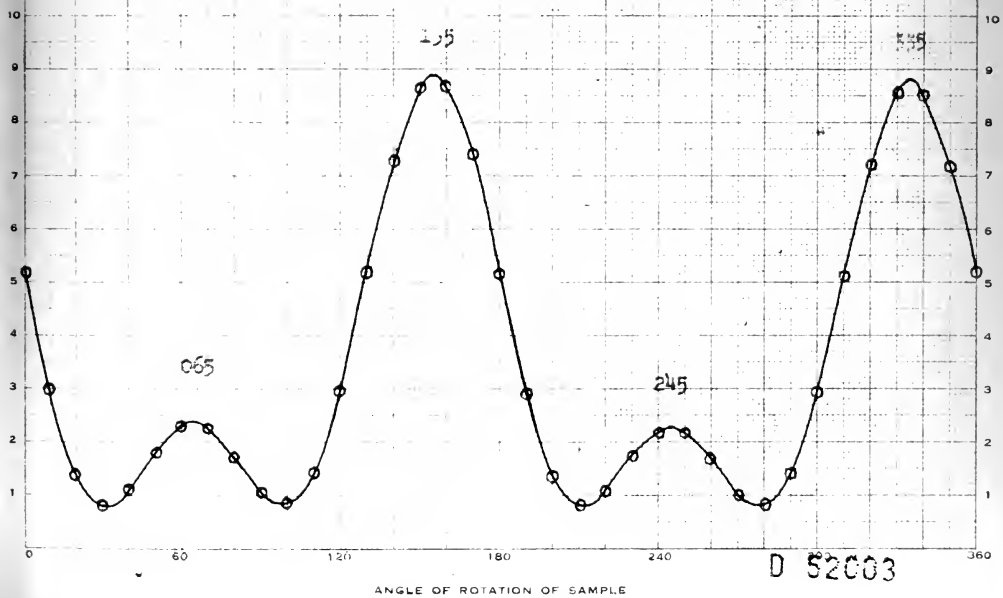
M_3	48.10	R_3	45.15
M_4	37.85	R_4	45.35
ΔM_{34}	10.25	ΔR_{34}	- .70

Av ΔM 10.30 PHASE SHIFT

Av ΔR - .65 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 10 113 PPM N_2 DATE 27 MAY 1952 PM

CRYSTAL No 1 SURFACE FILM CONDITION 8th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 287		@ 017		@ 107		@ 197	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	48.0	45.0	37.3	45.7	47.7	44.8	37.7	45.2
	48.0	45.0	37.3	45.8	47.5	44.9	37.6	45.8
Av.	48.00	45.00	37.30	45.75	47.60	44.85	37.65	45.50

M_1	48.00	R_1	45.00
M_2	37.30	R_2	45.75
ΔM_{12}	10.70	ΔR_{12}	- .75

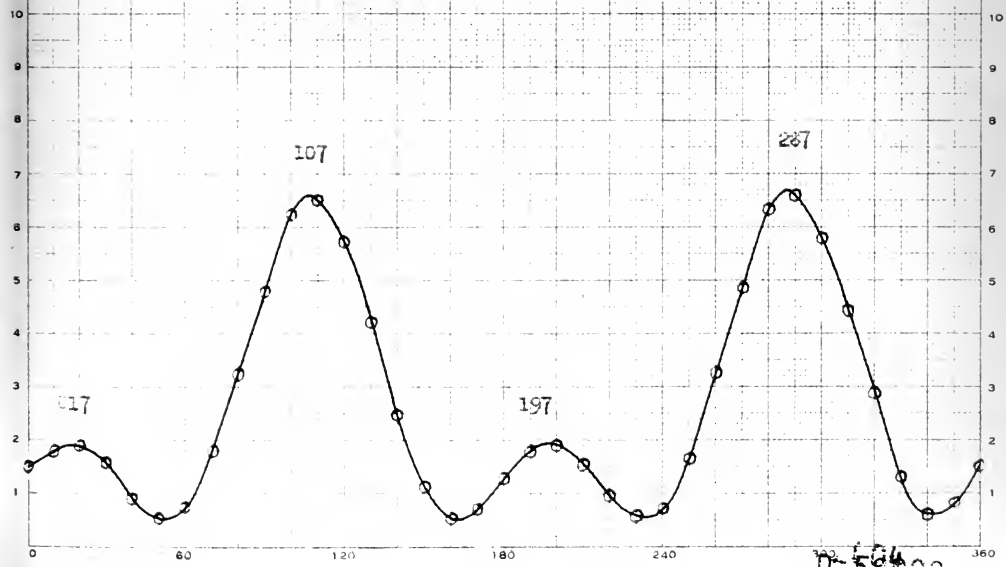
M_3	47.60	R_3	44.85
M_4	37.65	R_4	45.50
ΔM_{34}	9.95	ΔR_{34}	- .65

Av ΔM 10.33 PHASE SHIFT

Av ΔR - .70 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D-52003

SAMPLE No. 10 113 PPM N_2 DATE 29 MARCH 1952 FM

CRYSTAL No. 1 SURFACE FILM CONDITION 9th AUTOCLAVE OF 15 MIN. @ 233°C

@ 086		@ 176		@ 266		@ 356	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
49.6	45.0	38.2	45.8	49.0	44.9	37.4	46.0
49.0	45.2	37.6	46.0	49.2	45.1	37.4	45.8
49.30	45.10	37.90	45.90	49.10	45.00	37.40	45.90

M_1 49.30	R_1 45.10
M_2 37.90	R_2 45.90
ΔM_{12} 11.40	ΔR_{12} - .80

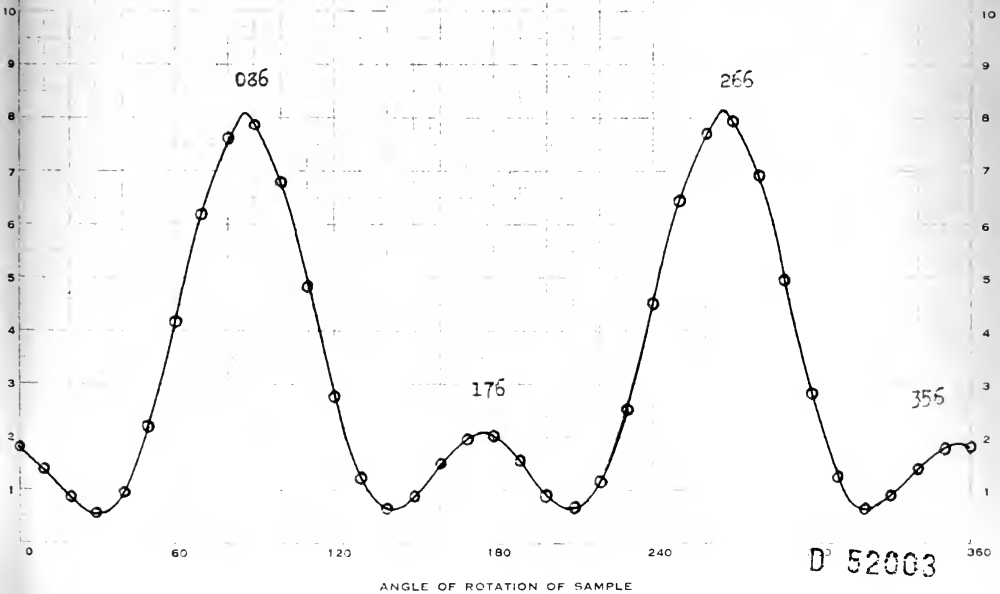
M_3 49.10	R_3 45.00
M_4 37.40	R_4 45.90
ΔM_{34} 11.70	ΔR_{34} - .90

Av ΔM 11.55 PHASE SHIFT

Av ΔR - .85 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



SAMPLE No. 10 — 113 PPM N_2 — DATE 30 MARCH 1952 PM

CRYSTAL No 1 — SURFACE FILM CONDITION 10th AUTOCLAVE OF 15 MIN. @ 233°C

@ 056		@ 146		@ 236		@ 326	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
49.1	45.2	37.5	46.4	48.8	44.9	38.0	45.8
49.0	45.4	37.9	46.2	48.4	45.3	38.0	45.9
V. 49.05	45.30	37.70	46.30	48.60	45.10	38.00	45.85

M_1	49.05	R_1	45.30
M_2	37.70	R_2	46.30
ΔM_{12}	11.35	ΔR_{12}	-1.00

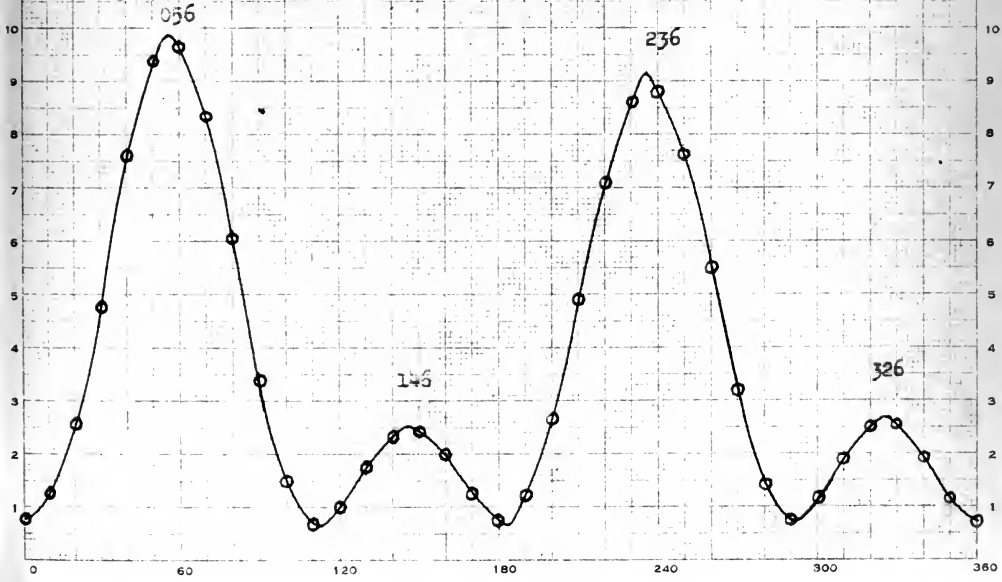
M_3	48.60	R_3	45.10
M_4	38.00	R_4	45.85
ΔM_{34}	10.60	ΔR_{34}	-.75

Av ΔM 10.98 PHASE SHIFT

Av ΔR -.88 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 10 113 PPM N_2 DATE 1 APRIL 1958 FN

CRYSTAL No 1 SURFACE FILM CONDITION 11th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 335		@ 245		@ 157		@ 067	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	45.0	45.0	36.1	45.7	47.3	45.2	36.1	46.1
	47.8	45.0	36.1	46.0	47.6	45.2	36.3	45.9
Av	47.90	45.00	36.10	45.85	47.45	45.20	36.20	46.00

M	47.90	R	45.00
M_2	36.10	R_2	45.85
ΔM_{12}	11.80	ΔR_{12}	- .85

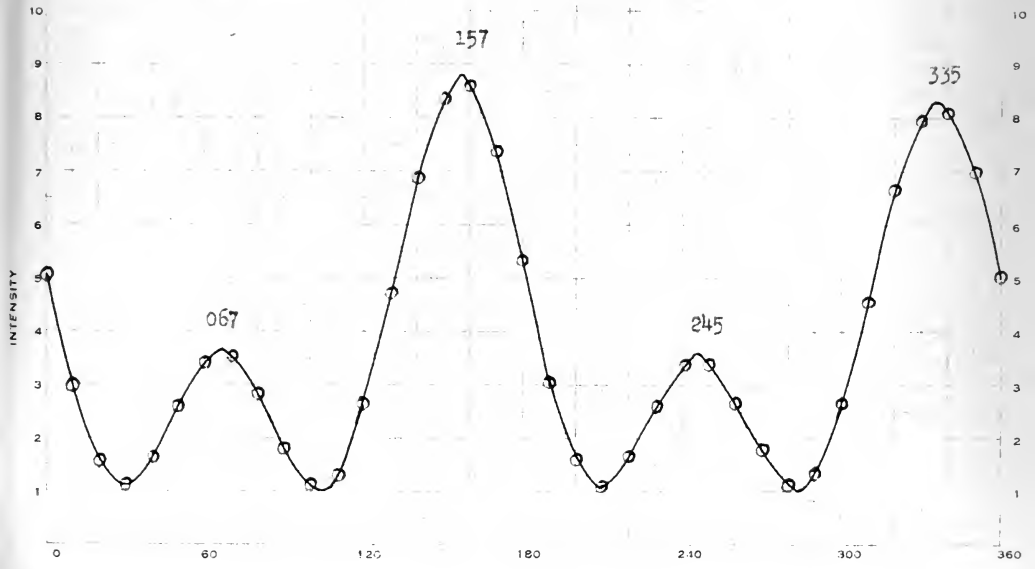
M_3	47.45	R_3	45.20
M_4	36.20	R_4	46.00
ΔM_{34}	11.25	ΔR_{34}	- .80

Av ΔM 11.53 PHASE SHIFT

Av ΔR - .83 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 10 113 PPM N_2 DATE 2 APRIL 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 12th AUTOCLAVE OF 15 MIN. @ 233°C

@ 277		@ 007		@ 097		@ 187	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
48.0	45.0	36.7	45.7	47.3	45.0	36.8	45.8
48.0	45.1	36.5	45.8	47.5	45.0	36.2	45.8
Av. 48.00	45.05	36.60	45.75	47.40	45.00	36.50	45.80

M_1	48.00	R_1	45.05
M_2	36.60	R_2	45.75
ΔM_{12}	11.40	ΔR_{12}	-.70

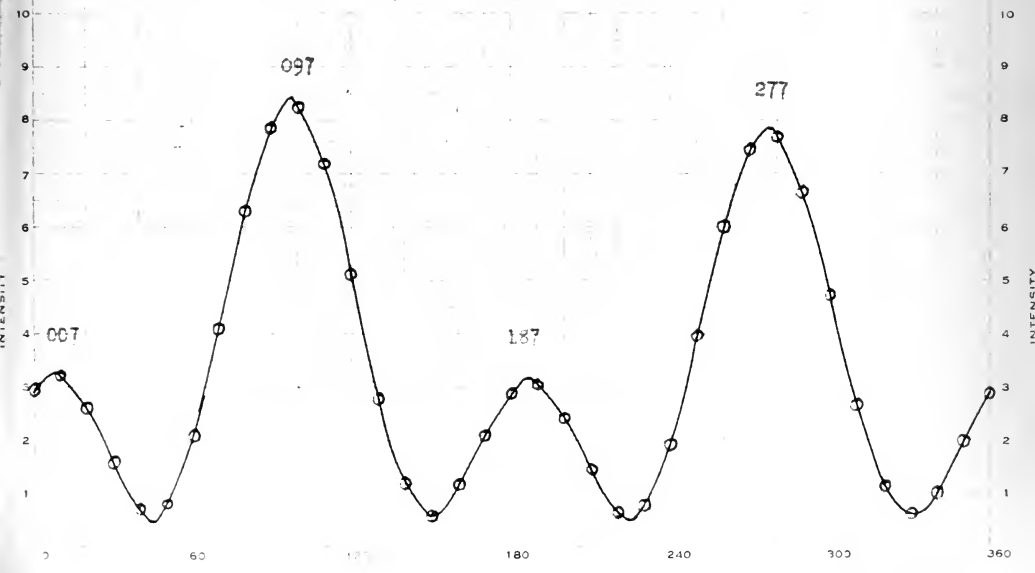
M_3	47.40	R_3	45.00
M_4	36.50	R_4	45.80
ΔM_{34}	10.90	ΔR_{34}	-.80

Av ΔM 11.15 PHASE SHIFT

Av ΔR -.75 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 10 113 PPM N_2 DATE 3 APRIL 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 13th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 057		@ 147		@ 237		@ 327	
	M_1	R_1	M_4	R_2	M_3	R_3	M_4	R_4
	47.8	44.6	36.4	46.0	47.9	44.8	36.2	45.7
	47.9	44.8	36.2	45.8	47.7	44.8	36.3	45.9
Av.	45.35	44.80	36.30	45.90	47.80	44.80	36.25	45.80

M_1	47.85	R_1	44.80
M_2	36.30	R_2	45.90
ΔM_{12}	11.55	ΔR_{12}	-1.10

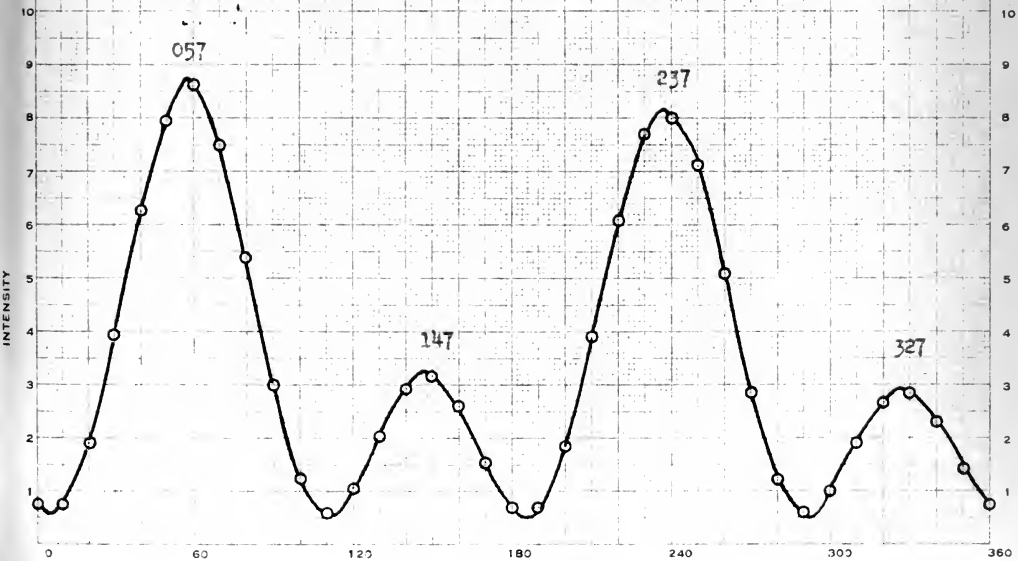
M_3	47.80	R_3	44.80
M_4	36.25	R_4	45.80
ΔM_{34}	11.55	ΔR_{31}	-1.00

Av ΔM 11.55 PHASE SHIFT

Av ΔR -1.05 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 10 113_{PPM} N_2 DATE 5 APRIL 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 15th AUTOCLAVE OF 15 MIN. @ 233°C

@ 358		@ 088		@ 178		@ 268	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
49.6	45.1	38.4	46.4	49.7	45.2	38.0	46.2
50.0	45.1	38.2	46.3	49.7	45.3	37.6	46.4
49.80	45.10	38.30	46.35	49.7	45.25	37.80	46.30

M_1 49.80 R_1 45.10
M_2 38.30 R_2 46.35
ΔM_{12} 11.50 ΔR_{12} -1.25

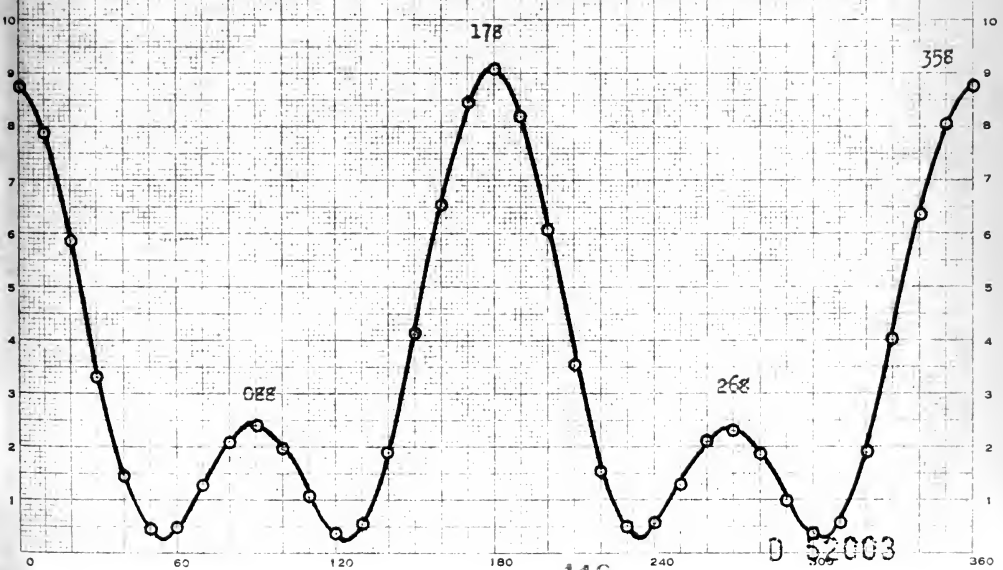
M_3 49.70 R_3 45.25
M_4 37.80 R_4 46.30
ΔM_{34} 11.90 ΔR_{34} -1.05

Av ΔM 11.70 PHASE SHIFT

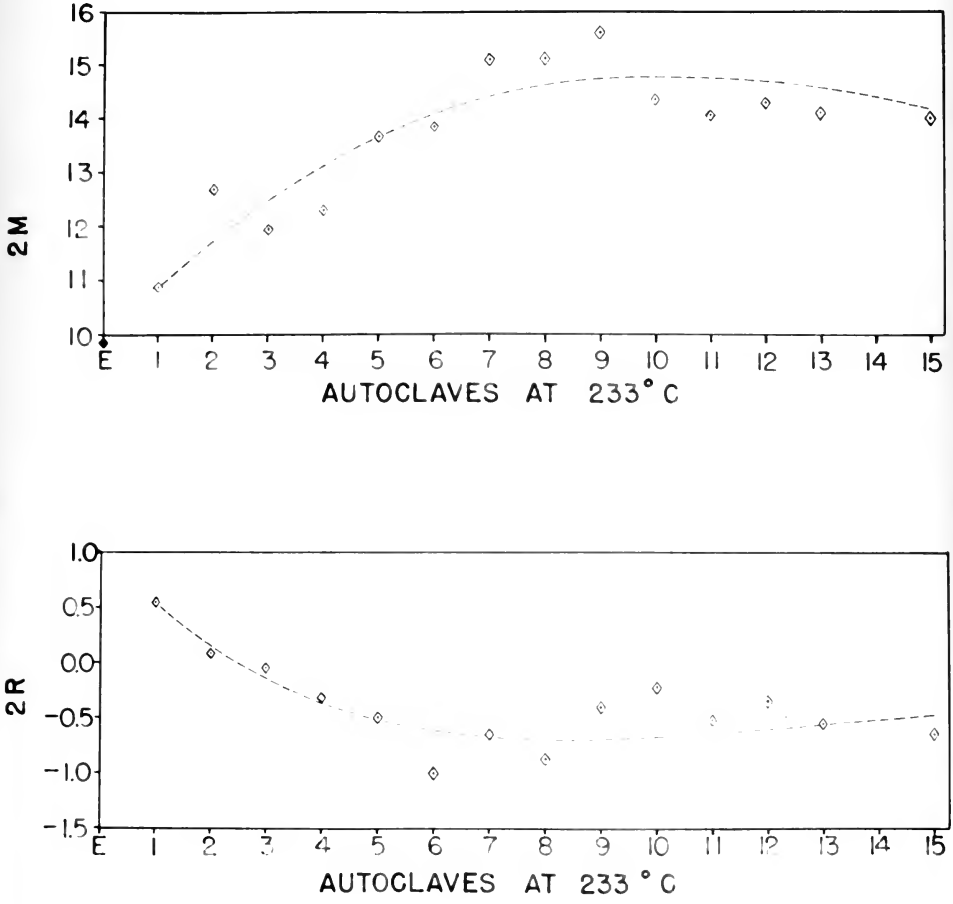
Av ΔR -1.15 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



PHASE SHIFT (2M) AND ROTATION OF PLANE OF POLARIZATION (2R) VS. CORROSION TIME FOR SAMPLE NO. ID-1

D 52203

SAMPLE No. 1D 147 PPM N_2 DATE 13 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 1st AUTOCLAVE 15 MIN. at 233°C

	@ 039		@ 309		@ 219		@ 129	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	48.2	45.7	37.3	45.2	48.1	45.8	37.3	45.1
	48.1	45.7	37.3	45.3	48.2	45.7	37.2	45.1
Av.	48.15	45.70	37.30	45.25	48.15	45.75	37.25	45.10

M_1	48.15	R_1	45.70
M_2	37.30	R_2	45.25
ΔM_{12}	10.85	ΔR_{12}	0.45

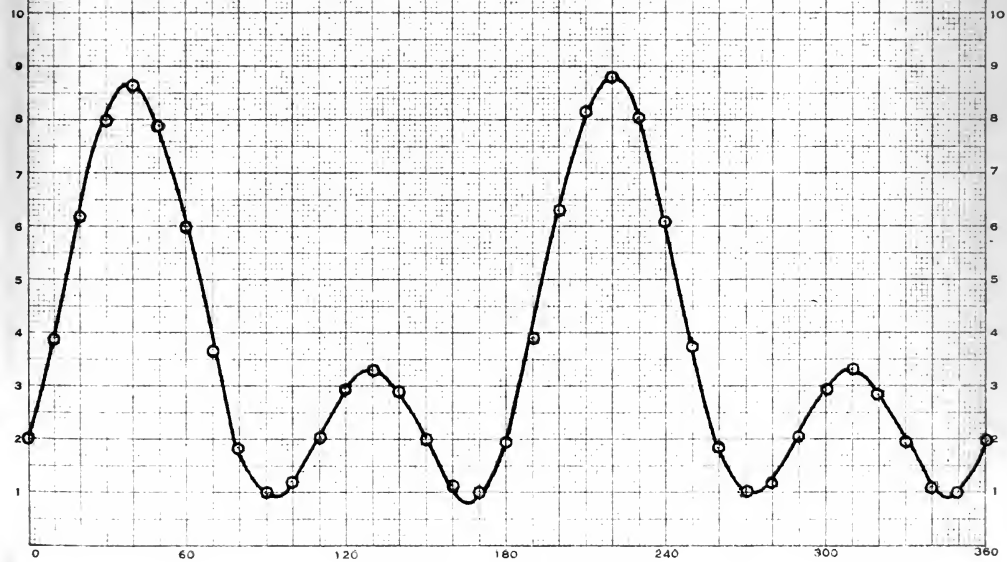
M_3	48.15	R_3	45.75
M_4	37.25	R_4	45.10
ΔM_{34}	10.90	ΔR_{34}	0.65

Av ΔM 10.87 PHASE SHIFT

Av ΔR 0.55 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1D 147 PPM N_2 DATE 14 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 2nd AUTOCLAVE 15 MIN. at 233°C

@ 294		@ 204		@ 114		@ 024	
M1	R1	M2	R2	M3	R3	M4	R4
49.2	45.7	36.3	45.5	49.6	45.3	36.7	45.3
49.5	45.7	36.8	45.5	49.5	45.3	36.6	45.4
Av. 49.35	45.70	36.85	45.50	49.55	45.30	36.65	45.35

M_1	49.35	R_1	45.70
M_2	36.85	R_2	45.50
ΔM_{12}	12.50	ΔR_{12}	0.20

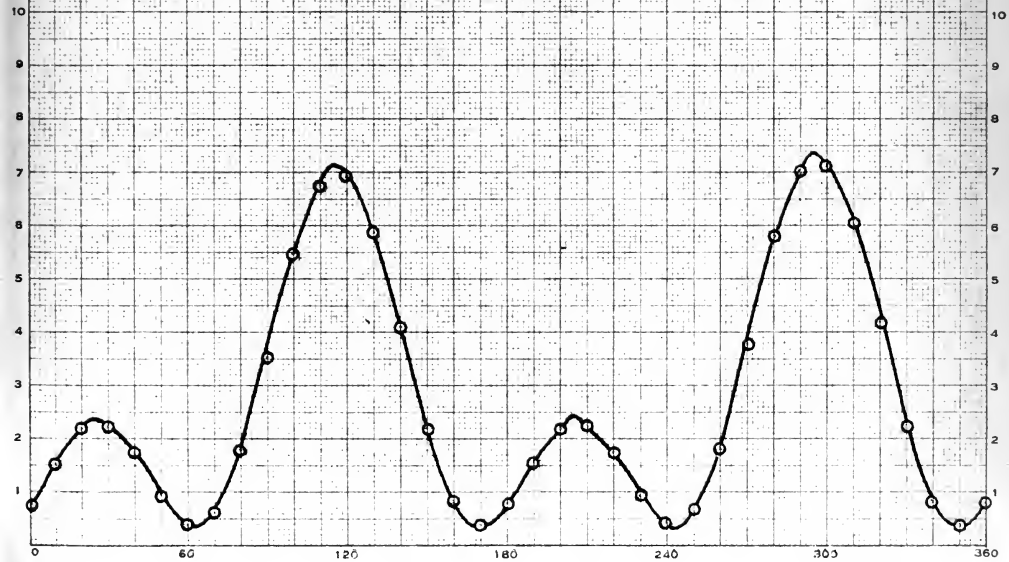
M_3	49.55	R_3	45.30
M_4	36.65	R_4	45.35
ΔM_{34}	12.90	ΔR_{34}	- .05

Av ΔM 12.70 PHASE SHIFT

Av ΔR .08 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1D 147 PPM N_2 DATE 17 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 3rd AUTOCLAVE 15 MIN. at 233°C

	@ 105		@ 215		@ 125		@ 035	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	48.7	45.5	36.4	45.7	48.7	45.7	36.9	45.5
	48.5	45.6	36.5	45.6	48.2	45.5	36.5	45.5
					48.5	45.6	36.8	45.8
Av.	48.60	45.55	36.45	45.65	48.47	45.60	36.73	45.60

M_1	48.60	R_1	45.55
M_2	36.45	R_2	45.65
ΔM_{12}	12.15	ΔR_{12}	- .10

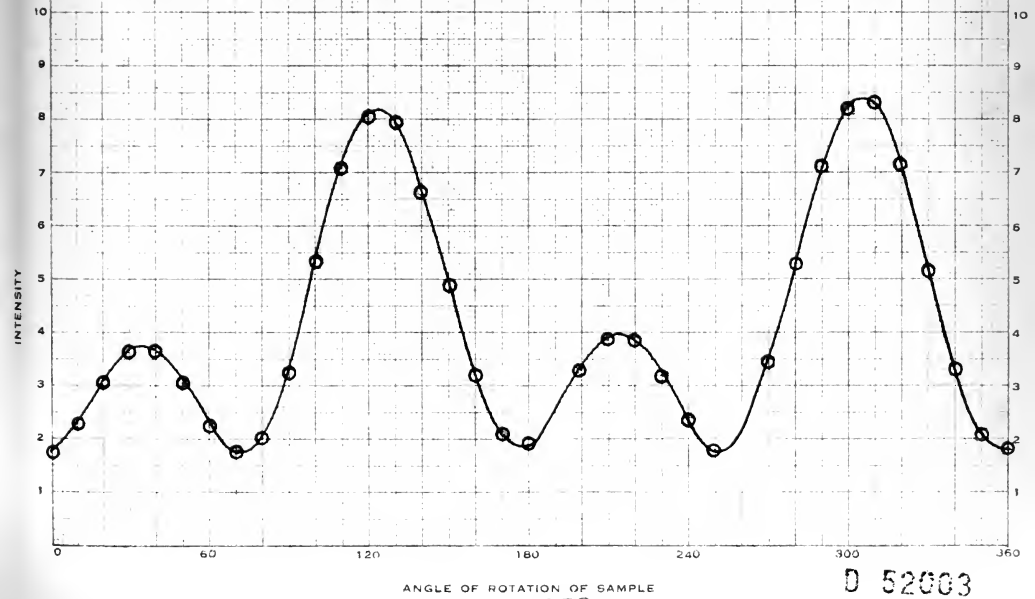
M_3	48.47	R_3	45.60
M_4	36.73	R_4	45.60
ΔM_{34}	11.74	ΔR_{34}	0.00

Av ΔM 11.95 PHASE SHIFT

Av ΔR -0.05 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1D 147 PPM N_2 DATE 22 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 4th AUTOCLAVE 15 MIN. @ 233°C

	@ 013		@ 283		@ 193		@ 103	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	47.8	45.3	35.7	45.7	47.9	45.3	35.7	45.6
	48.2	45.3	35.5	45.8	47.7	45.5	35.5	45.6
Av.	48.00	45.30	35.60	45.75	47.80	45.40	35.60	45.60

M_1	48.00	R_1	45.30
M_2	35.60	R_2	45.75
ΔM_{12}	12.40	ΔR_{12}	-0.45

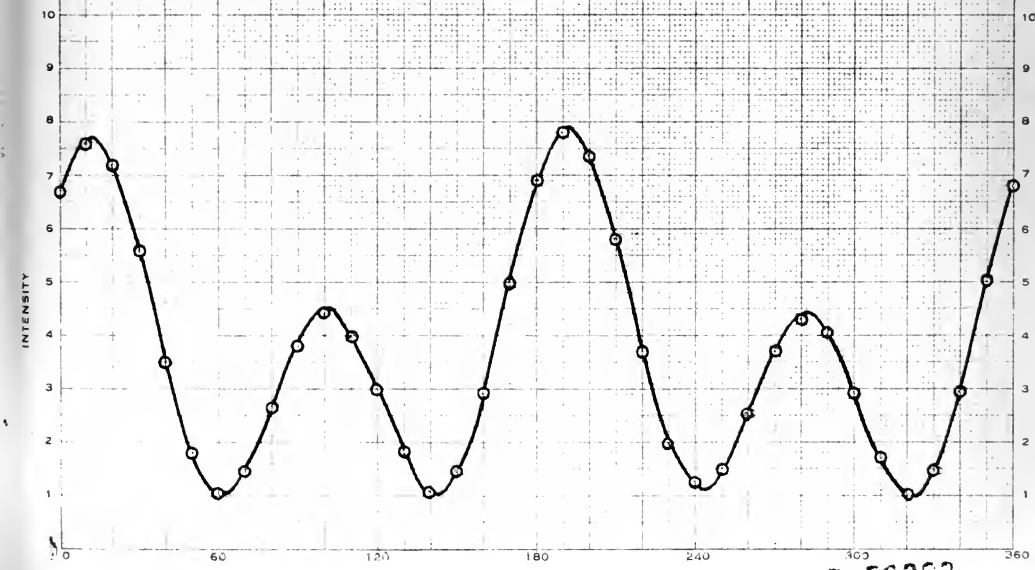
M_3	47.80	R_3	45.40
M_4	35.60	R_4	45.60
ΔM_{34}	12.20	ΔR_{34}	-0.20

Av ΔM 12.30 PHASE SHIFT

Av ΔR -0.32 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1D 147 PPM N_2 DATE 24 MARCH 1952 PM

CRYSTAL No 1 SURFACE FILM CONDITION 5th ANNEALED 15 MIN. @ 233°C

@ 353		@ 263		@ 173		@ 083	
M1	R1	M2	R2	M3	R3	M4	R4
47.9	45.3	34.2	45.8	47.9	45.4	34.2	45.8
47.8	45.3	34.3	45.7	47.9	45.3	34.1	45.8
Av. 47.85	45.30	34.25	45.75	47.90	45.35	34.15	45.80

M_1	47.85	R_1	45.30
M_2	34.25	R_2	45.75
ΔM_{12}	13.60	ΔR_{12}	-0.45

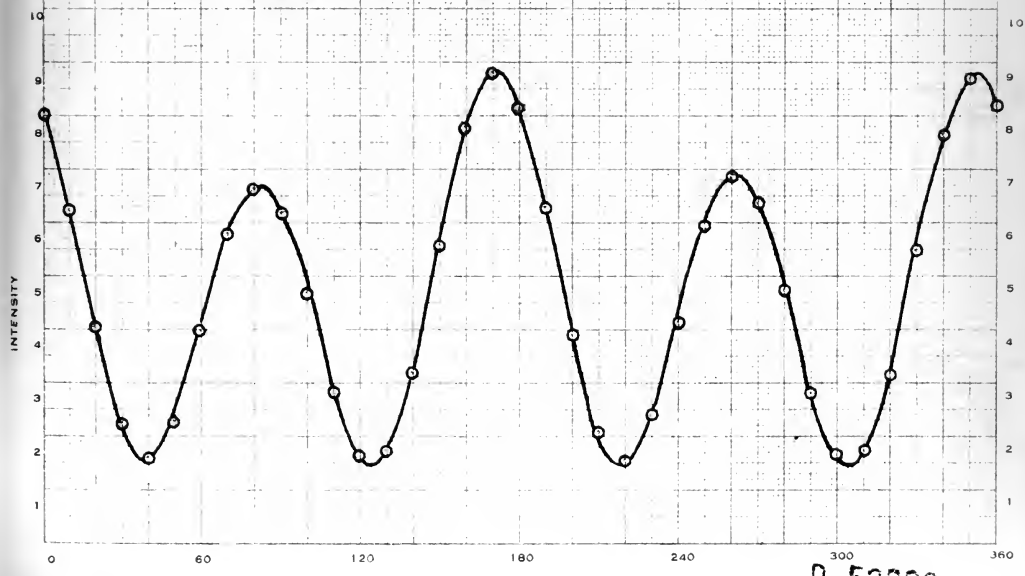
M_3	47.90	R_3	45.35
M_4	34.15	R_4	45.80
ΔM_{34}	13.75	ΔR_{34}	-0.55

Av ΔM 13.67 PHASE SHIFT

Av ΔR -0.50 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1D 147ppm N_2 DATE 25 MARCH 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 6th AUTOCLAVE 15 MIN. @ 233°C

	@ 346		@ 256		@ 166		@ 076	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	50.3	45.2	36.6	46.3	50.3	45.5	36.4	46.5
	50.3	45.2	36.5	46.2	50.4	45.6	36.4	46.5
Av.	50.30	45.20	36.55	46.25	50.35	45.55	36.40	46.50

M_1 50.30 R_1 45.20
M_2 36.55 R_2 46.25
ΔM_{12} 13.75 ΔR_{12} -1.05

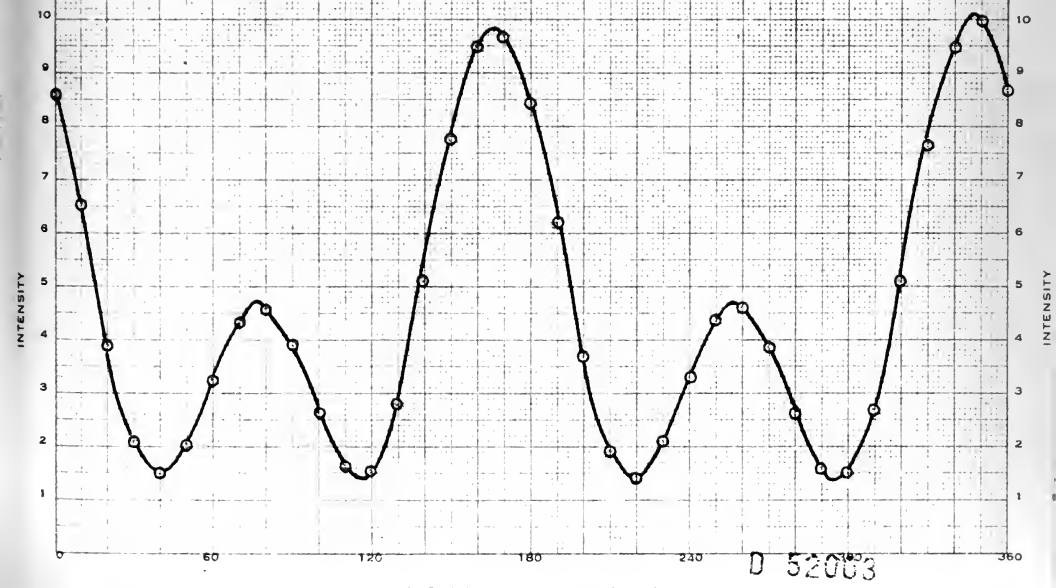
M_3 50.35 R_3 45.55
M_4 36.40 R_4 46.50
ΔM_{34} 13.95 ΔR_{34} -0.95

Av ΔM 13.85 PHASE SHIFT

Av ΔR -1.00 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1D 147 PPM N_2 DATE 26 MARCH 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 7th AUTOCLAVE 15 MIN. @ 233°C

	@ 331		@ 241		@ 151		@ 061	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	50.5	45.4	35.3	46.0	50.2	45.4	35.2	46.1
	50.6	45.3	35.3	46.1	50.3	45.5	35.4	46.0
Av.	50.55	45.35	35.30	46.05	50.25	45.45	35.30	46.05

M_1	50.55	R_1	45.35
M_2	35.30	R_2	46.05
ΔM_{12}	15.25	ΔR_{12}	-0.70

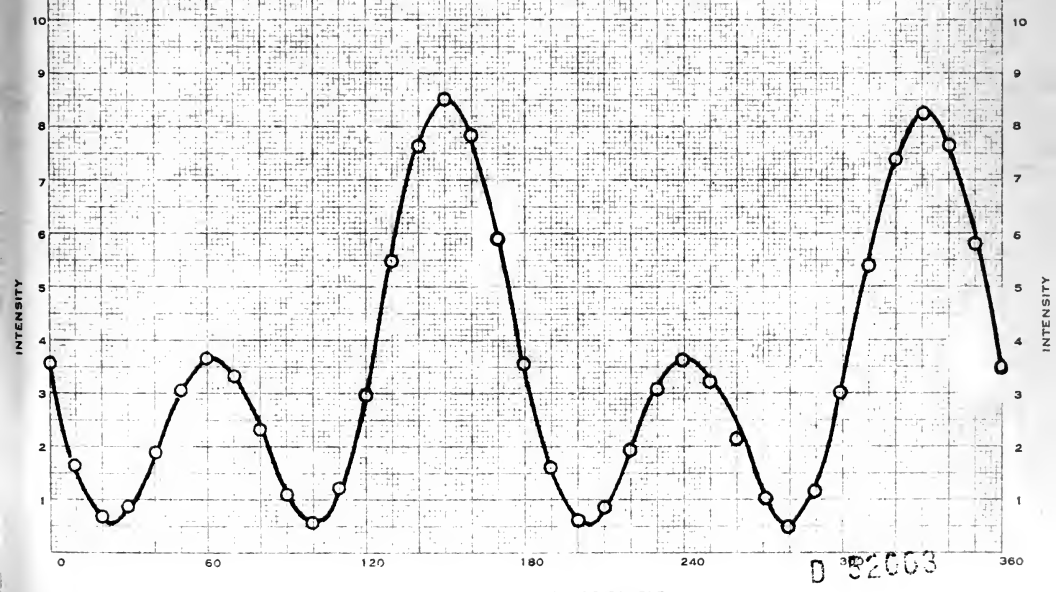
M_3	50.25	R_3	45.45
M_4	35.30	R_4	46.05
ΔM_{34}	14.95	ΔR_{34}	-0.60

Av ΔM 15.10 PHASE SHIFT

Av ΔR -0.65 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 92003

SAMPLE No. 1D 147 PPM H_2 DATE 27 MARCH 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 8th AUTOCLAVE 15 MIN. @ 233°C

	@ 006		@ 276		@ 186		@ 096	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	52.2	45.0	37.1	46.1	51.6	45.1	36.6	45.8
	52.2	45.1	36.9	45.9	51.6	45.1	36.5	46.0
Av.	52.20	45.05	37.00	46.00	51.60	45.10	36.55	45.90

M_1	52.20	R_1	45.05
M_2	37.00	R_2	46.00
ΔM_{12}	15.20	ΔR_{12}	-0.95

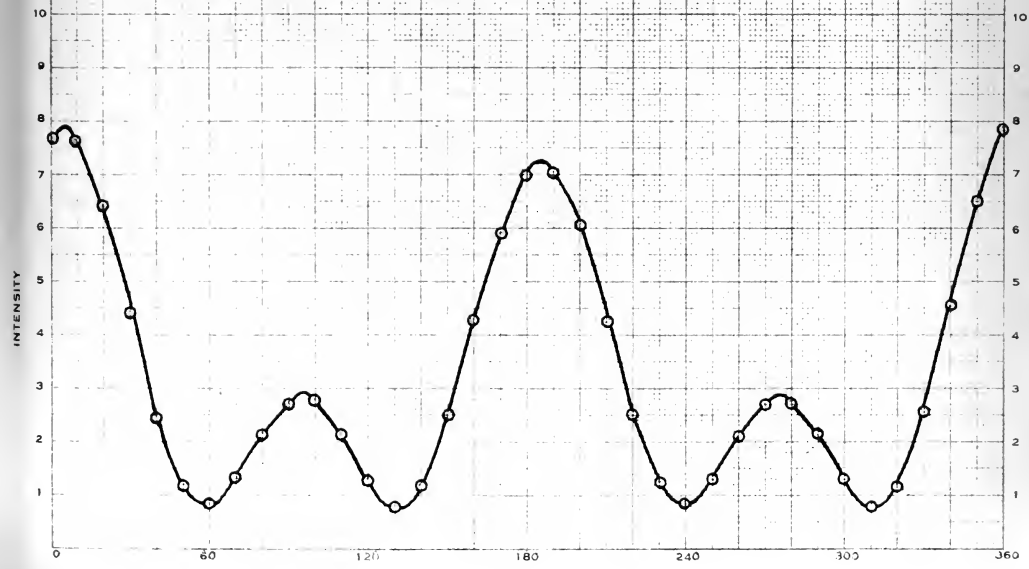
M_3	51.60	R_3	45.10
M_4	36.55	R_4	45.90
ΔM_{34}	15.05	ΔR_{34}	-0.80

Av ΔM 15.12 PHASE SHIFT

Av ΔR -0.87 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1D 147 PPM N2 DATE 29 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 9th AUTOCLAVE 15 MIN @ 233°C

	@ 316		@ 226		@ 136		@ 046	
	M1	R1	M2	R2	M3	R3	M4	R4
	52.4	45.6	36.7	46.0	51.9	45.7	36.4	46.0
	52.5	45.5	37.0	46.1	51.9	45.7	36.2	46.0
Av.	52.45	45.55	36.85	46.05	51.90	45.70	36.30	46.00

M_1	52.45	R_1	45.55
M_2	36.85	R_2	46.05
ΔM_{12}	15.60	ΔR_{12}	-0.50

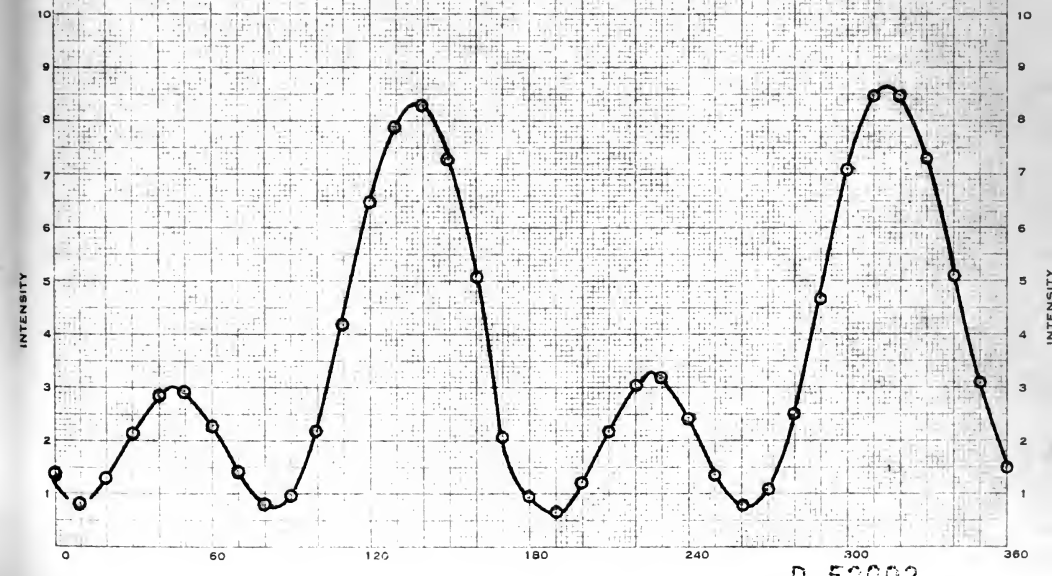
M_3	51.90	R_3	45.70
M_4	36.30	R_4	46.00
ΔM_{34}	15.60	ΔR_{34}	-0.30

Av ΔM 15.60 PHASE SHIFT

Av ΔR -0.40 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1D 147 PPM H_2 DATE 31 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 10th AUTOCLAVE 15 MIN. @ 233°C

	@ 337		@ 247		@ 157		@ 067	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	50.6	45.8	36.5	46.1	50.5	45.6	36.0	45.6
	50.3	45.7	36.2	46.0	50.5	45.5	35.7	45.8
Av.	50.45	45.75	36.35	46.05	50.50	45.55	35.85	45.70

M_1	50.45	R_1	45.75
M_2	36.35	R_2	46.05
ΔM_{12}	14.10	ΔR_{12}	-.30

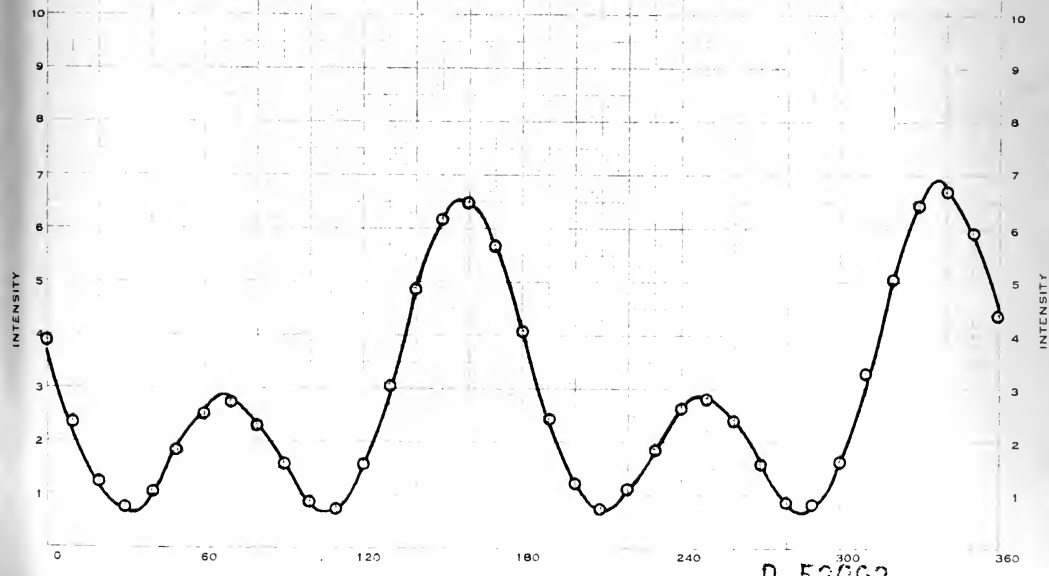
M_3	50.50	R_3	45.55
M_4	35.85	R_4	45.70
ΔM_{34}	14.65	ΔR_{34}	-.15

Av ΔM 14.37 PHASE SHIFT

Av ΔR - .22 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1D 147 PPM N_2 DATE 1 APRIL 1952 AM

CRYSTAL No 1 SURFACE FILM CONDITION 11th AUTOCLAVE 15 MIN. @ 233°C

	@ 274		@ 184		@ 094		@ 004	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	50.4	45.1	36.0	45.6	49.7	45.2	35.7	45.9
	50.1	45.1	36.3	45.6	49.8	45.3	35.7	45.7
Av.	50.25	45.10	36.15	45.60	49.75	45.25	35.70	45.80

M_1	50.25	R_1	45.10
M_2	36.15	R_2	45.60
ΔM_{12}	14.10	ΔR_{12}	-.50

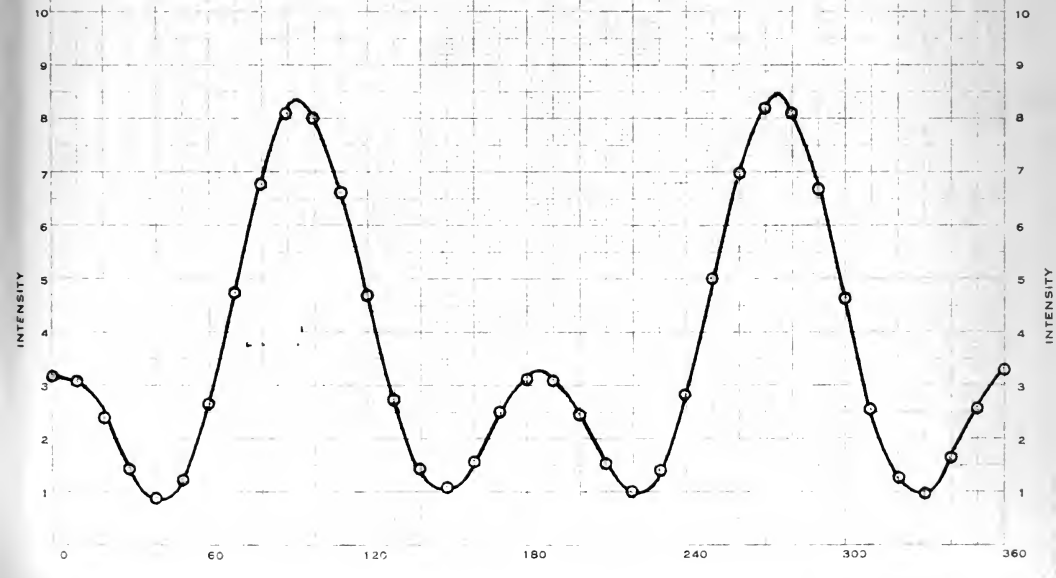
M_3	49.75	R_3	45.25
M_4	35.70	R_4	45.80
ΔM_{34}	14.05	ΔR_{34}	-.55

Av ΔM 14.07 PHASE SHIFT

Av ΔR -.52 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1D 147 PPM N_2 DATE 2 APRIL 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 12th AUTOCLAVE 15 MIN. @ 233°C

	@ 203		@ 113		@ 023		@ 293	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	49.1	45.3	35.0	45.8	49.5	45.2	34.7	45.5
	49.3	45.3	35.3	45.7	49.3	45.4	35.0	45.6
Av.	49.20	45.30	35.15	45.75	49.40	45.30	34.85	45.55

M_1	49.20	R_1	45.30
M_2	35.15	R_2	45.75
ΔM_{12}	14.05	ΔR_{12}	-.45

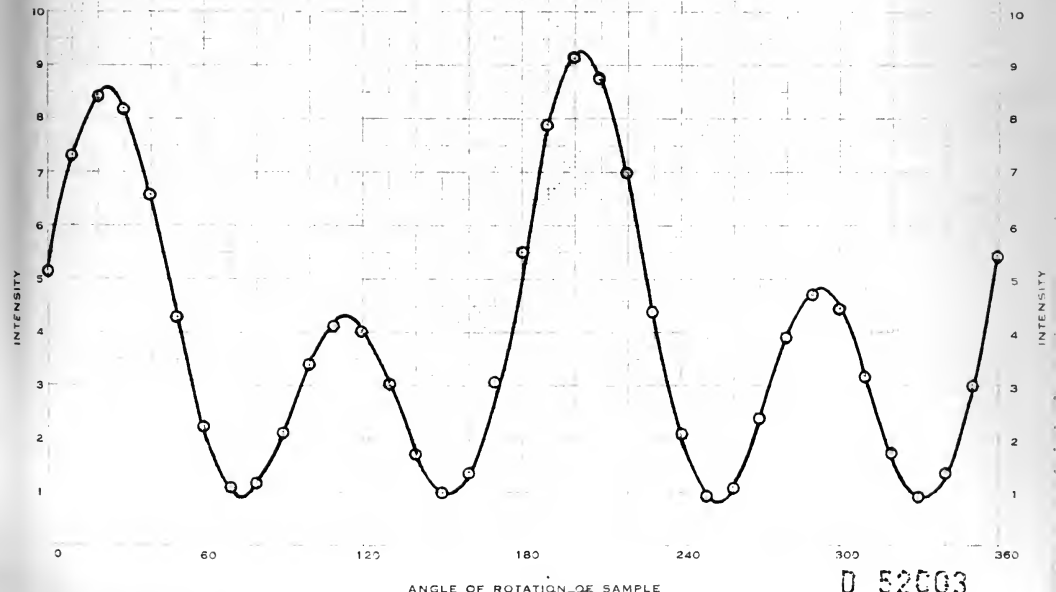
M_3	49.40	R_3	45.30
M_4	34.85	R_4	45.55
ΔM_{34}	14.55	ΔR_{34}	-.25

Av ΔM 14.30 PHASE SHIFT

Av ΔR -.35 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1D 147 PPM N_2 DATE 4 APRIL 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 13th AUTOCLAVE 15 MIN. @ 233°C

	@ 216		@ 126		@ 036		@ 306	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	49.8	45.4	35.4	45.8	49.2	45.8	35.0	46.4
	49.4	45.8	35.4	46.3	49.2	45.5	35.3	46.2
	49.5	45.5						
Av.	49.57	45.57	35.40	46.05	49.20	45.65	35.15	46.30

M_1 49.57 R_1 45.57

M_2 35.40 R_2 46.05

ΔM_{12} 14.17 ΔR_{12} −.48

M_3 49.20 R_3 45.65

M_4 35.15 R_4 46.30

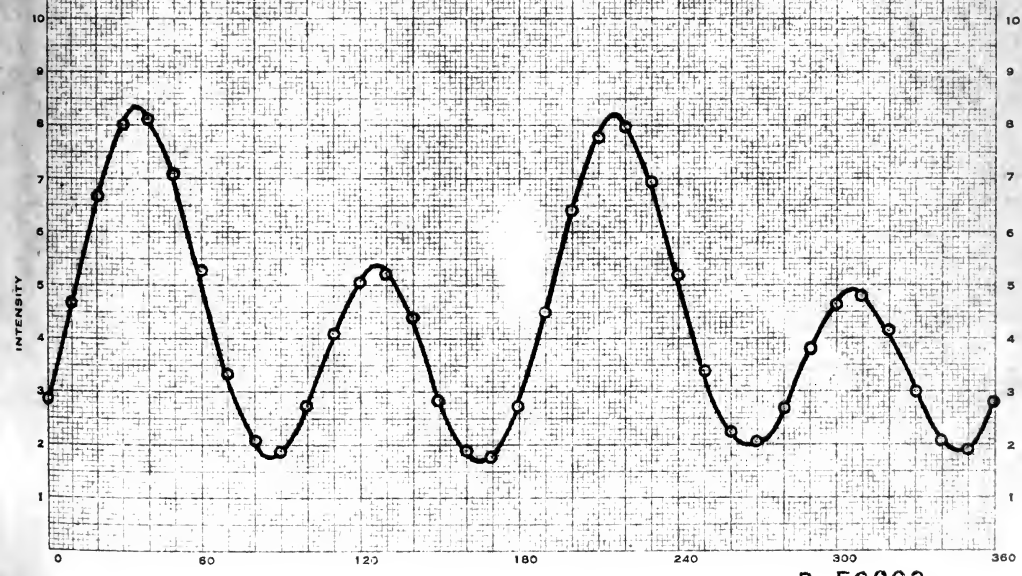
ΔM_{34} 14.05 ΔR_{34} −.65

Av ΔM 14.11 PHASE SHIFT

Av ΔR −0.56 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1D 147 PPM N_2 DATE 5 APRIL 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 15th AUTOCLAVE of 15 MIN. @ 233°C

	@ 336		@ 246		@ 156		@ 066	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	51.4	45.7	37.7	46.50	51.1	45.50	36.8	46.1
	51.2	45.7	37.6	46.40	51.0	45.70	36.6	46.2
Av.	51.30	45.70	37.65	46.45	51.05	45.60	36.70	46.15

M_1	51.30	R_1	45.70
M_2	37.65	R_2	46.45
ΔM_{12}	13.65	ΔR_{12}	- .75

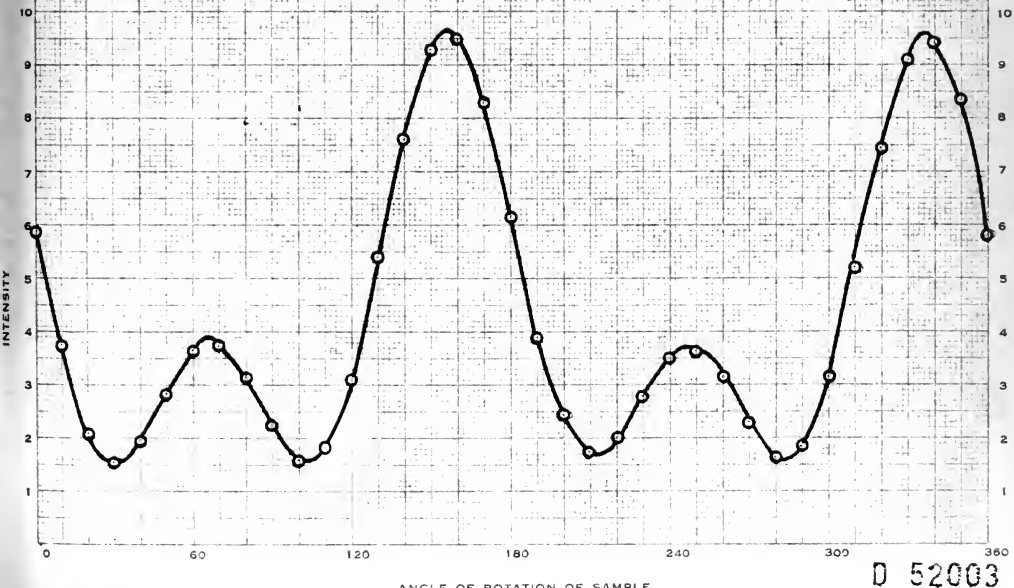
M_3	51.05	R_3	45.60
M_4	36.70	R_4	46.15
ΔM_{34}	14.35	ΔR_{34}	- .55

Av ΔM 14.00 PHASE SHIFT

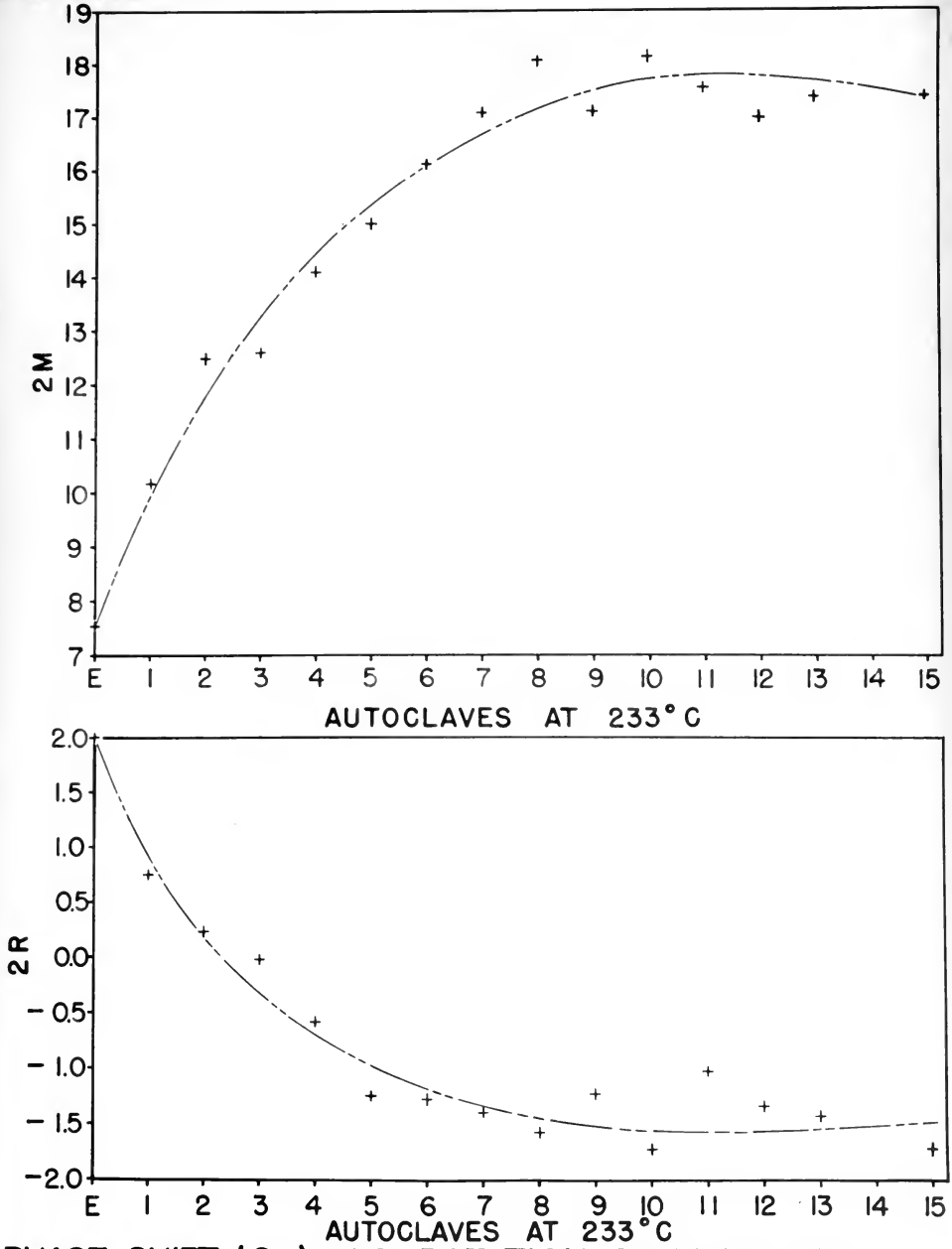
Av ΔR -0.65 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



PHASE SHIFT (2m) AND ROTATION OF PLANE OF POLARIZATION (2r) VS. CORROSION TIME FOR SAMPLE NO. 1E - 1

SAMPLE No 1E 150 PPM N_2 DATE 6 March 1952 PM

CRYSTAL No 1 SURFACE FILM CONDITION AS ELECTROPOLISHED

	@ 014		@ 104		@ 194		@ 284	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	47.1	46.4	39.0	44.2	46.4	46.5	39.3	44.6
	46.9	46.5	39.4	44.4	46.8	46.3	39.3	44.5
Av.	47.00	46.45	39.20	44.30	46.60	46.40	39.30	44.55

M_1	47.00	R_1	46.45
M_2	39.20	R_2	44.30
ΔM_{12}	7.80	ΔR_{12}	2.15

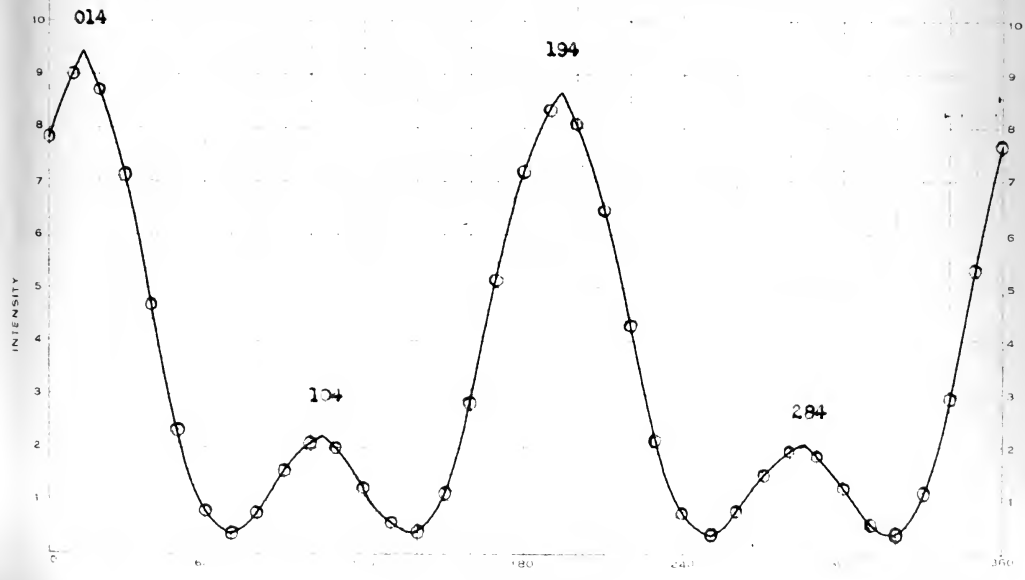
M_3	46.60	R_3	46.40
M_4	39.30	R_4	44.55
ΔM_{34}	7.30	ΔR_{34}	1.85

Av ΔM 7.55 PHASE SHIFT

Av ΔR 2.00 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. PPM DATE

CRYSTAL No. SURFACE FILM CONDITION

@	M1	R1	M2	R2	M3	R3	M4	R4
	46.7	46.5	36.3	46.	45.8	45.1	36.6	45.0
	46.0	45.0	36.8	45.2	46.3	46.0	36.5	45.6
Av.	46.35	45.95	35.55	45.05	46.20	45.40	36.55	45.30

M_1	46.35	R_1	45.95
M_2	36.55	R_2	45.05
ΔM_{12}	9.80	ΔR_{12}	.90

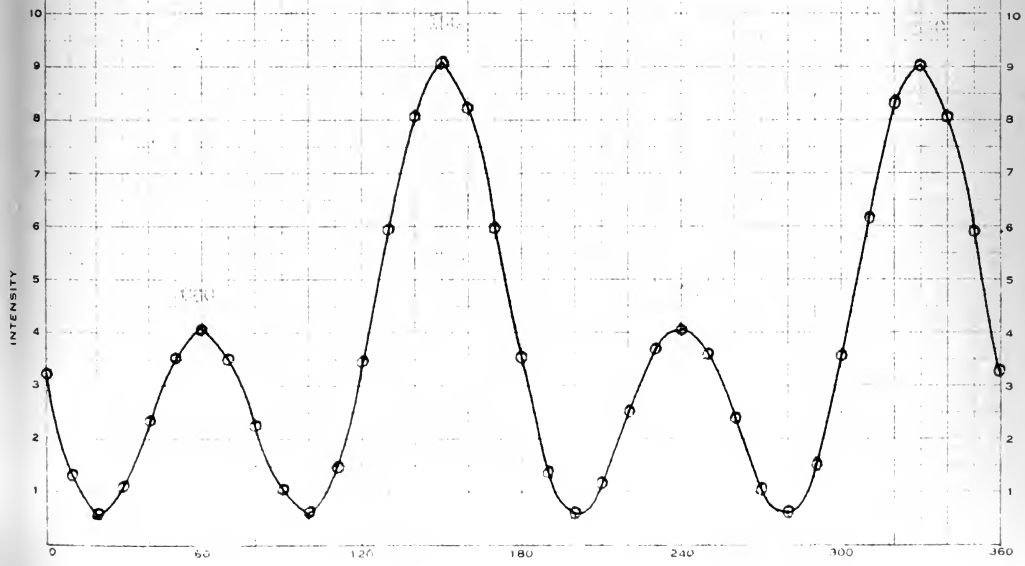
M_3	46.20	R_3	45.80
M_4	36.55	R_4	45.30
ΔM_{34}	9.65	ΔR_{34}	.50

Av ΔM 9.73 PHASE SHIFT

Av ΔR .75 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No PPM DATE

CRYSTAL No 3 SURFACE FILM CONDITION

	@ 330		@ 006		@ 150		@ 240	
	M1	R1	M2	R2	M3	R3	M4	R4
	49.2	45.5	50.6	45.4	49.3	46.0	36.9	45.2
	49.2	45.7	38.5	45.3	49.2	45.7	35.9	45.6
Av.	49.20	45.50	38.55	45.30	49.25	45.85	36.35	45.40

M_1	49.20	R_1	45.50
M_2	38.55	R_2	45.30
ΔM_{12}	10.65	ΔR_{12}	-.10

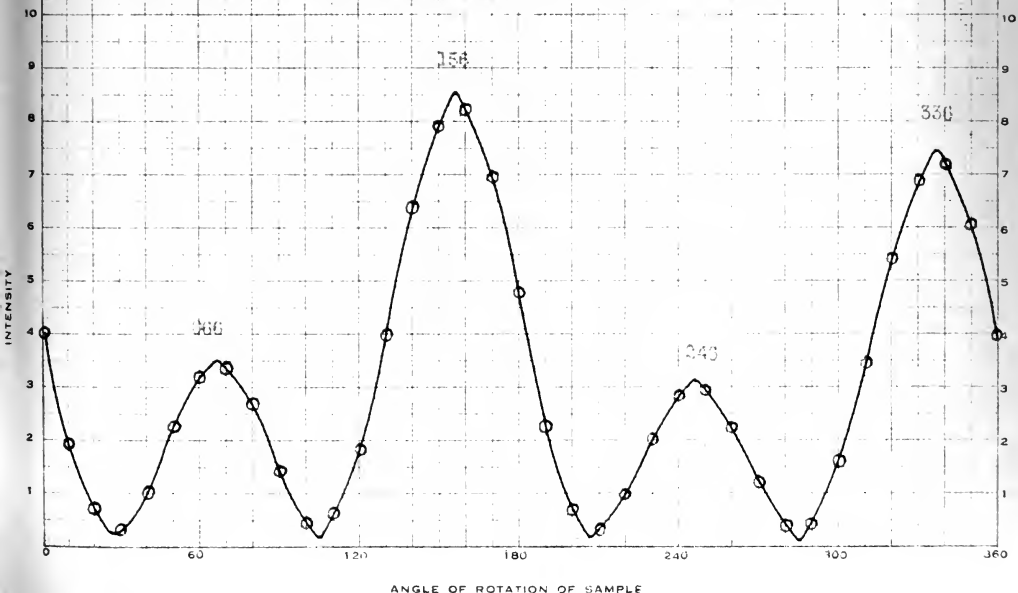
M_3	49.25	R_3	45.85
M_4	36.95	R_4	45.40
ΔM_{34}	12.30	ΔR_{34}	.45

Av ΔM 11.48 PHASE SHIFT

Av ΔR .28 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



SAMPLE No. [illegible] PPM ~ DATE [illegible]

CRYSTAL No 1 SURFACE FILM CONDITION

@ 078		@ 168		@ 258		@ 348	
M1	R1	M2	R2	M3	R3	M4	R4
49.1	45.4	34.4	45.4	49.0	45.8	36.5	45.5
49.0	45.5	36.6	45.5	49.2	45.4	36.3	45.5
Av. 49.05	45.45	35.50	45.50	49.10	45.60	36.40	45.50

M_1	49.05	R_1	45.45
M_2	35.50	R_2	45.50
ΔM_{12}	13.55	ΔR_{12}	-.05

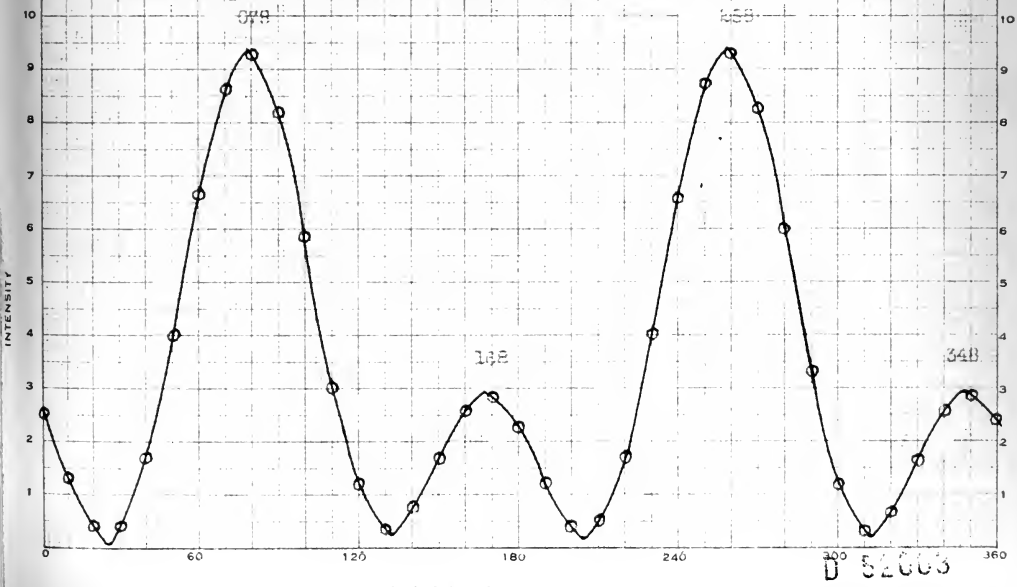
M_3	49.00	R_3	45.60
M_4	36.40	R_4	45.50
ΔM_{34}	12.60	ΔR_{34}	0

Av ΔM 13.58 PHASE SHIFT

Av ΔR -.02 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1E 150 PPM N_2 DATE 22 MARCH 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 4th AUTOCLAVE OF 15 MIN. at 233°C

	@ 005		@ 095		@ 185		@ 275	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	48.8	45.5	34.7	46.1	48.5	45.5	34.7	46.0
	48.9	45.5	34.9	46.2	48.6	45.5	34.3	46.0
Av.	48.85	45.50	34.80	46.15	48.60	45.50	34.50	46.00

M_1	48.85	R_1	45.50
M_2	34.80	R_2	46.15
ΔM_{12}	14.05	ΔR_{12}	- .65

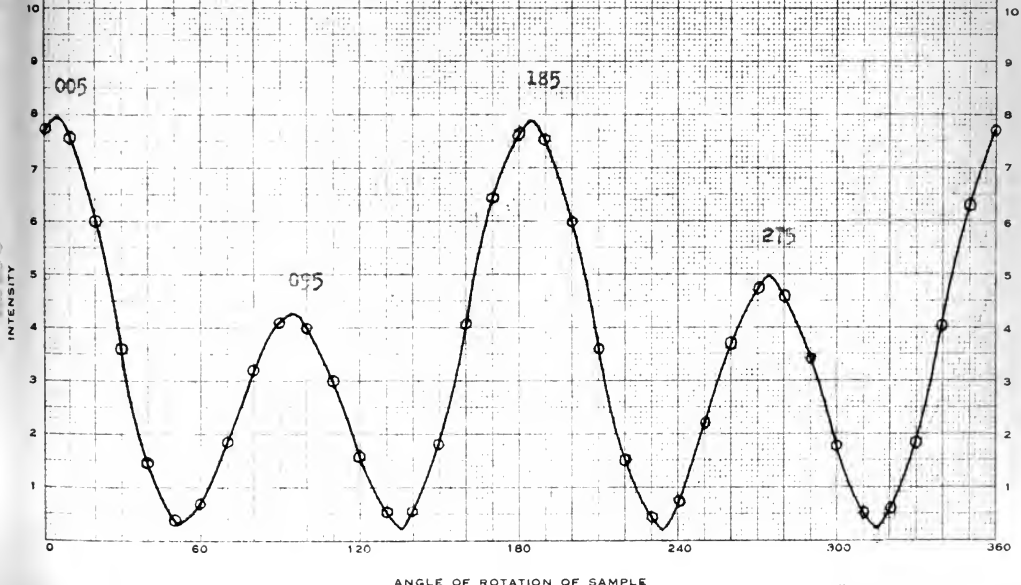
M_3	48.60	R_3	45.50
M_4	34.50	R_4	46.00
ΔM_{34}	14.10	ΔR_{34}	- .50

Av ΔM 14.08 PHASE SHIFT

Av ΔR - .58 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 12 [illegible] PPM [illegible] DATE 24 MARCH 1952 [illegible]

CRYSTAL No 1 SURFACE FILM CONDITION 5th AUTOCLAVE OF 15 MIN. at [illegible]

@ 028		@ 118		@ 208		@ 298	
M1	R1	M2	R2	M3	R3	M4	R4
49.3	45.1	34.0	46.6	49.6	44.9	34.4	46.2
49.3	45.3	34.5	46.5	49.3	45.4	34.7	46.4
Av. 49.30	45.20	34.25	46.55	49.45	45.15	34.55	46.30

M_1	49.30	R_1	45.20
M_2	34.25	R_2	46.55
ΔM_{12}	15.05	ΔR_{12}	-1.35

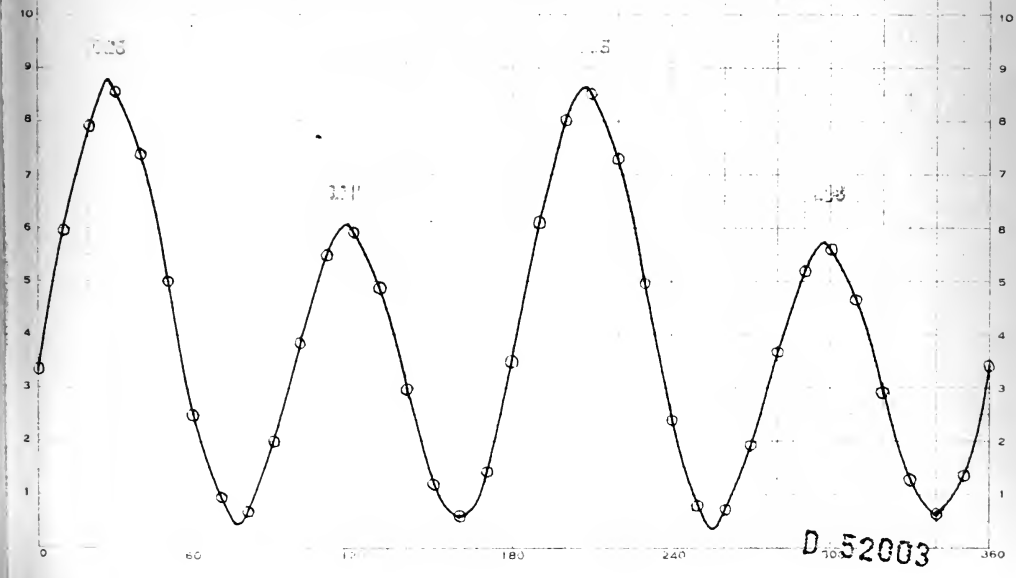
M_3	49.45	R_3	45.15
M_4	34.55	R_4	46.30
ΔM_{34}	14.90	ΔR_{34}	-1.15

Av ΔM 14.98 PHASE SHIFT

Av ΔR -1.25 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 1X 150 PPM N_2 DATE 25 MARCH [illegible]

CRYSTAL No [illegible] SURFACE FILM CONDITION [illegible]

	@ [illegible]		@ 099		@ [illegible]		@ 279	
	M	R	M_2	R_2	M_3	R_3	M_4	R_4
	51.3	45.0	34.8	46.4	50.7	44.9	35.1	46.2
	51.3	45.0	34.8	45.8	50.8	44.8	35.0	46.4
Av.	51.30	45.00	34.80	46.10	50.75	44.85	35.05	46.30

M_1	51.30	R_1	45.00
M_2	34.80	R_2	46.10
ΔM_{12}	16.50	ΔR_{12}	-1.10

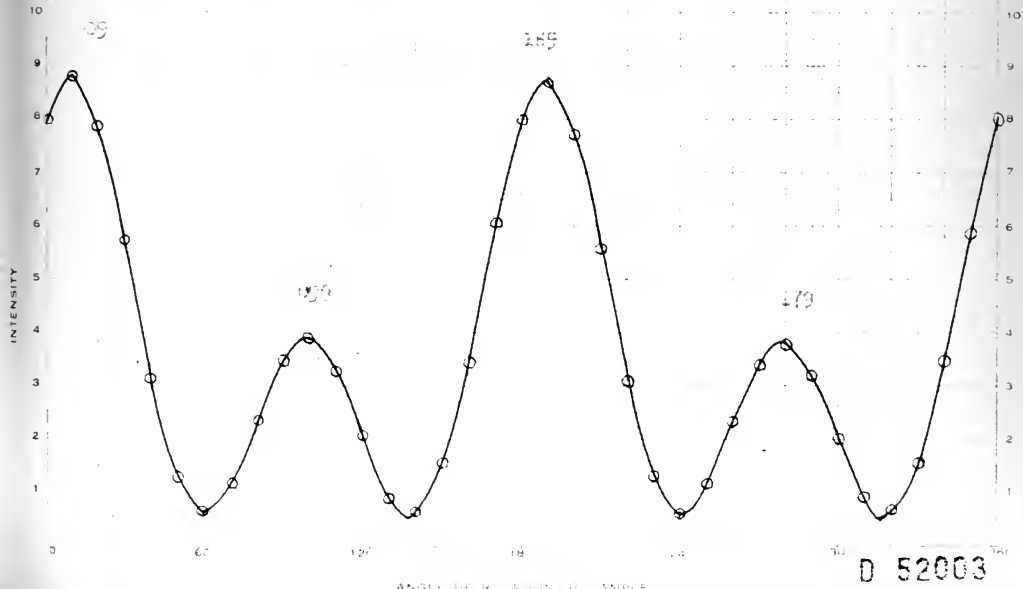
M_3	50.75	R_3	44.85
M_4	35.05	R_4	46.30
ΔM_{34}	15.70	ΔR_{34}	-1.45

Av ΔM 16.10 PHASE SHIFT

Av ΔR -1.28 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1E 150 PPM N_2 DATE 25 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 7th AUTOCLAVE OF 15 MIN. at 233°C

	@ 043		@ 133		@ 223		@ 313	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	51.9	44.9	34.7	46.1	52.0	44.2	34.8	45.9
	51.8	44.8	34.8	46.1	51.9	44.6	35.0	46.0
Av.	51.85	44.85	34.75	46.10	51.95	44.40	34.90	45.95

M_1	51.85	R_1	44.85
M_2	34.75	R_2	46.10
ΔM_{12}	17.10	ΔR_{12}	-1.25

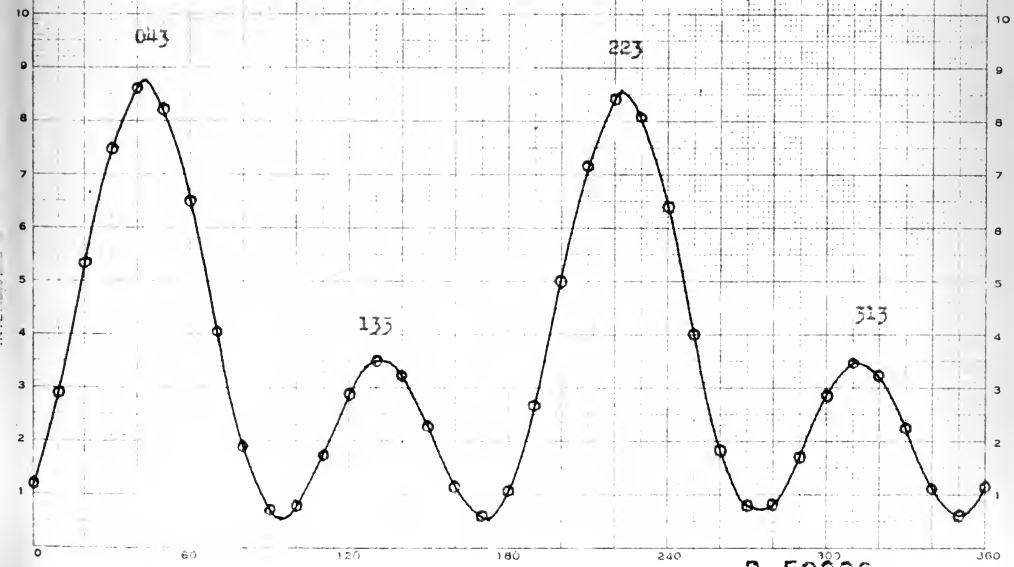
M_3	51.95	R_3	44.40
M_4	34.90	R_4	45.95
ΔM_{34}	17.05	ΔR_{34}	-1.55

Av ΔM 17.08 PHASE SHIFT

Av ΔR -1.40 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 1E 150 PPM N_2 DATE 27 MARCH 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 8th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 081		@ 171		@ 261		@ 351	
	M1	R1	M2	R2	M3	R3	M4	R4
	51.9	44.5	33.4	46.3	51.3	44.6	33.7	46.0
	51.7	44.9	33.6	46.2	51.4	44.6	33.3	46.4
Av.	51.80	44.70	33.50	46.25	51.35	44.60	33.50	46.20

M_1	51.80	R_1	44.70
M_2	33.50	R_2	46.25
ΔM_{12}	18.30	ΔR_{12}	-1.55

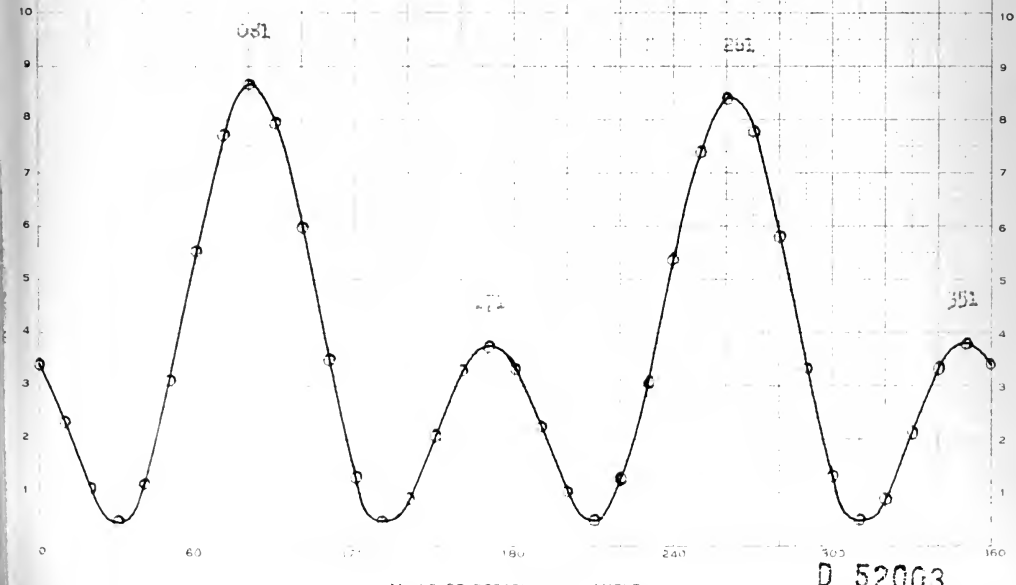
M_3	51.35	R_3	44.60
M_4	33.50	R_4	46.20
ΔM_{34}	17.80	ΔR_{34}	-1.60

Av ΔM 18.05 PHASE SHIFT

Av ΔR -1.58 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1E 150 PPM N_2 DATE 29 MARCH 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 9th AUTOCLAVE OF 15 MIN. @ 233°C

@ 330		@ 060		@ 150		@ 240	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
52.9	44.8	35.7	45.9	52.2	45.1	35.2	46.0
52.6	44.6	35.5	46.2	51.9	45.0	34.8	46.3
Av. 52.75	44.70	35.60	46.05	52.05	45.05	35.00	46.15

M_1	52.75	R_1	44.70
M_2	35.60	R_2	46.05
ΔM_{12}	17.15	ΔR_{12}	-1.35

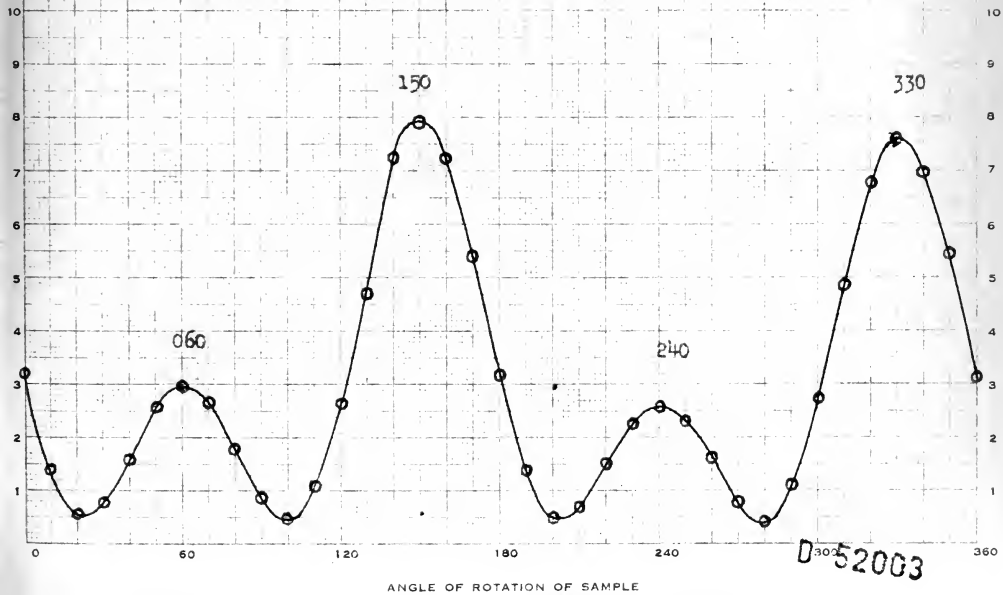
M_3	52.05	R_3	45.05
M_4	35.00	R_4	46.15
ΔM_{34}	17.05	ΔR_{34}	-1.10

Av ΔM 17.10 PHASE SHIFT

Av ΔR -1.23 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



SAMPLE No. 13 150 PPM N_2 DATE 30 MARCH 1952 PM

CRYSTAL No 1 SURFACE FILM CONDITION 10th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 031		@ 121		@ 211		@ 301	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	52.3	45.0	34.6	46.6	51.9	44.6	33.7	46.2
	52.5	44.8	34.2	46.6	52.0	44.6	33.7	46.5
Av.	52.40	44.90	34.40	46.60	51.95	44.60	33.70	46.35

M_1 52.40 R_1 44.90
M_2 34.40 R_2 46.60
ΔM_{12} 18.00 ΔR_{12} -1.70

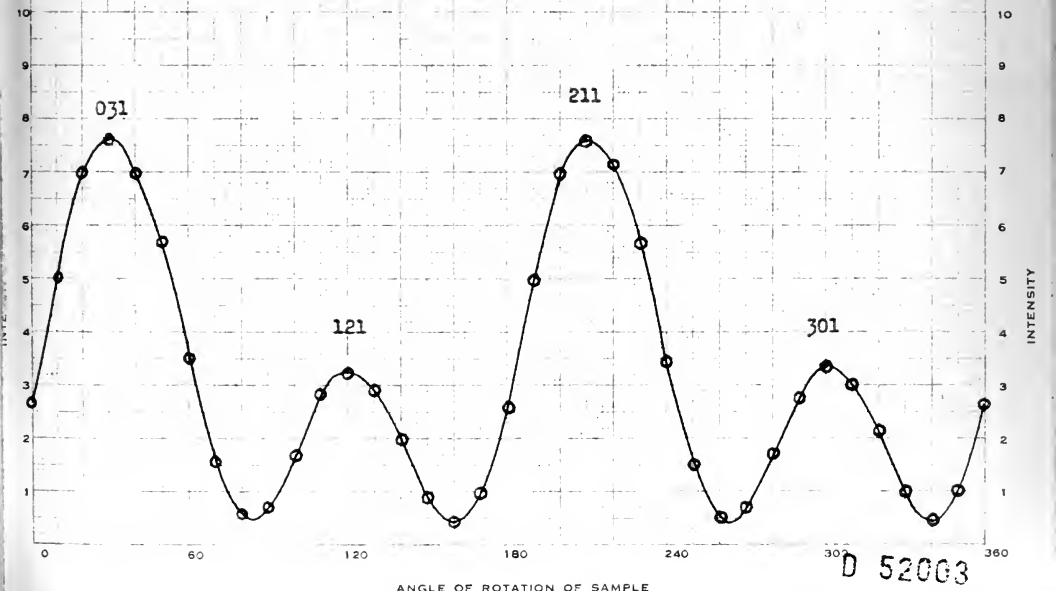
M_3 51.95 R_3 44.60
M_4 33.70 R_4 46.35
ΔM_{34} 18.25 ΔR_{31} -1.75

Av ΔM 18.13 PHASE SHIFT

Av ΔR -1.73 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1E 150 PPM N_2 DATE 1 APRIL 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 11th AUTOCLAVE OF 15 MIN. @ 233°C

@ 343		@ 073		@ 163		@ 253	
M1	R1	M2	R2	M3	R3	M4	R4
51.2	45.0	33.7	46.1	50.4	45.1	33.5	46.0
51.2	45.0	33.4	46.0	51.0	44.8	33.0	45.9
Av. 51.20	45.00	33.55	46.05	50.70	44.95	33.25	45.95

M_1	51.20	R_1	45.00
M_2	33.55	R_2	46.05
ΔM_{12}	17.65	ΔR_{12}	-1.05

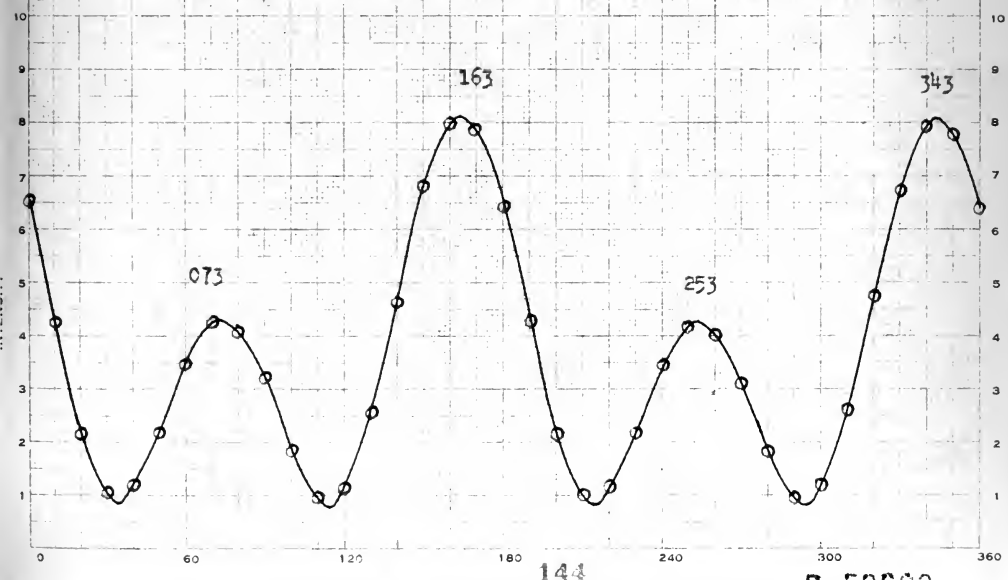
M_3	50.70	R_3	44.95
M_4	33.25	R_4	45.95
ΔM_{34}	17.45	ΔR_{34}	-1.00

Av ΔM 17.55 PHASE SHIFT

Av ΔR -1.03 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 12 150 PPM N_2 - DATE 2 APRIL 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 12th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 328		@ 058		@ 148		@ 238	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	50.7	44.7	33.6	46.3	50.0	44.9	33.0	46.0
	50.7	44.8	33.5	46.2	49.6	44.7	33.0	46.0
Av.	50.70	44.75	33.55	46.25	49.80	44.80	33.00	46.00

M_1	50.70	R_1	44.75
M_2	33.55	R_2	46.25
ΔM_{12}	17.15	ΔR_{12}	-1.50

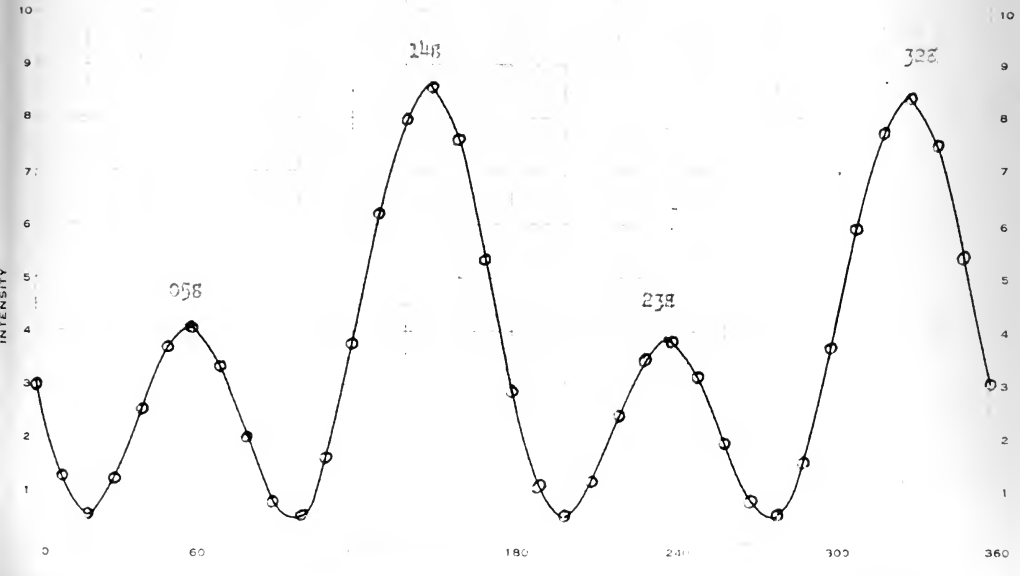
M_3	49.80	R_3	44.80
M_4	33.00	R_4	46.00
ΔM_{34}	16.80	ΔR_{34}	-1.20

Av ΔM 16.98 PHASE SHIFT

Av ΔR -1.35 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 14 150 PPM N_2 DATE 3 APRIL 1952 PM

CRYSTAL No 1 SURFACE FILM CONDITION 13th AUTOCLAVE OF 15 MIN. at 233°C

	@ 056		@ 146		@ 236		@ 326	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	51.2	45.2	33.7	46.2	50.4	44.8	33.4	46.3
	51.2	44.8	33.7	46.5	50.8	44.9	33.4	46.4
Av.	51.20	45.00	33.70	46.35	50.60	44.85	33.40	46.35

M_1	51.20	R_1	45.00
M_2	33.70	R_2	46.35
ΔM_{12}	17.50	ΔR_{12}	-1.35

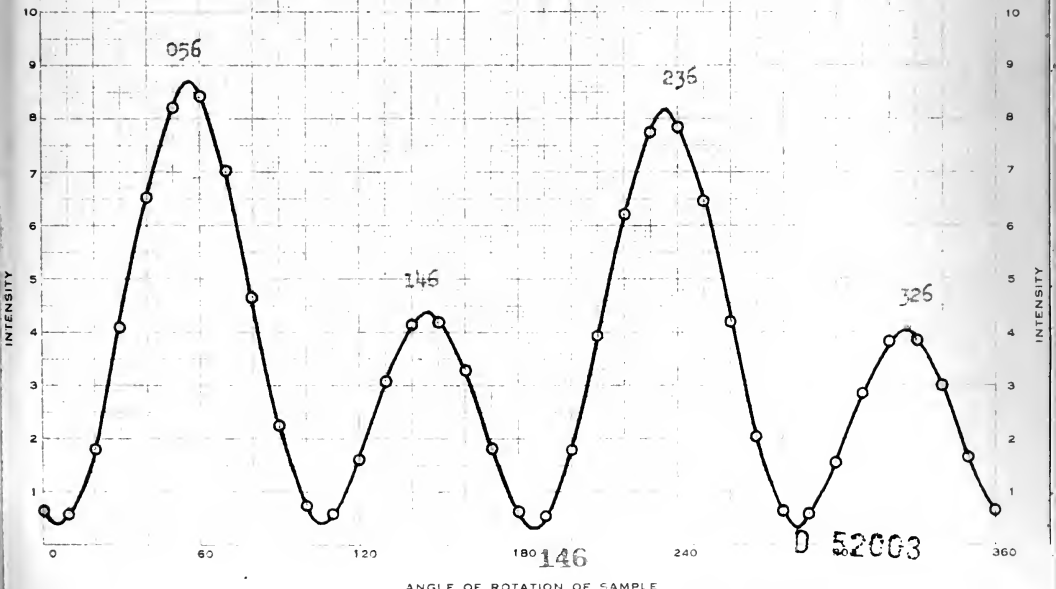
M_3	50.60	R_3	44.85
M_4	33.40	R_4	46.35
ΔM_{34}	17.20	ΔR_{34}	-1.50

Av ΔM 17.35 PHASE SHIFT

Av ΔR -1.43 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



146

D 52003

SAMPLE No. 13 · 150 PPM N_2 · DATE 5 APRIL 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 15th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 063		@ 153		@ 243		@ 333	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	52.2	44.5	34.6	46.3	51.3	44.7	34.5	46.2
	52.4	44.7	34.6	46.4	51.8	44.5	34.5	46.4
Av.	52.30	44.60	34.60	46.35	51.55	44.60	34.50	46.30

M_1	52.30	R_1	44.60
M_2	34.60	R_2	46.35
ΔM_{12}	17.70	ΔR_{12}	-1.75

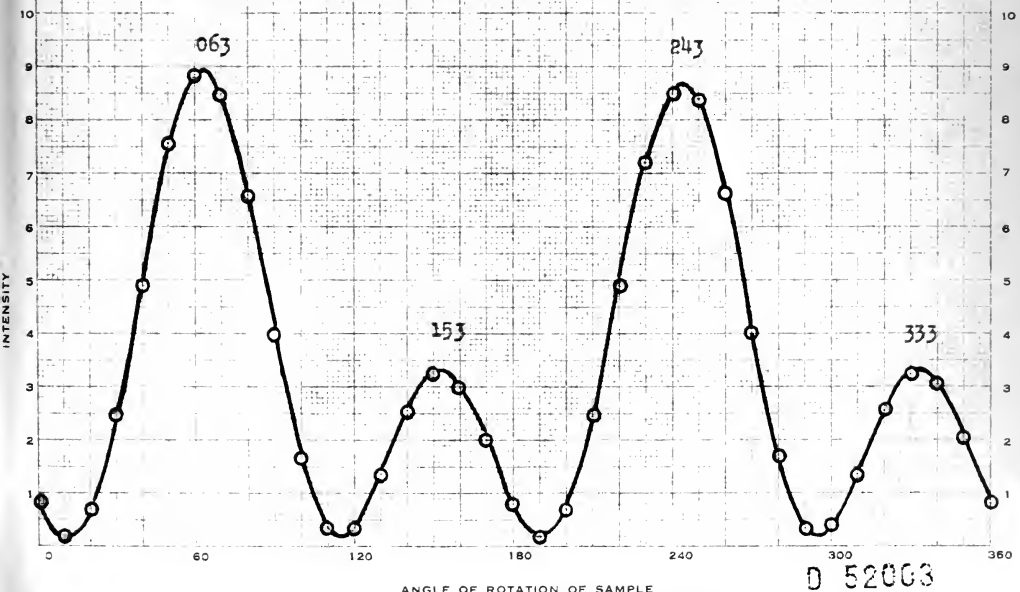
M_3	51.55	R_3	44.60
M_4	34.50	R_4	46.30
ΔM_{34}	17.05	ΔR_{34}	-1.70

Av ΔM 17.38 PHASE SHIFT

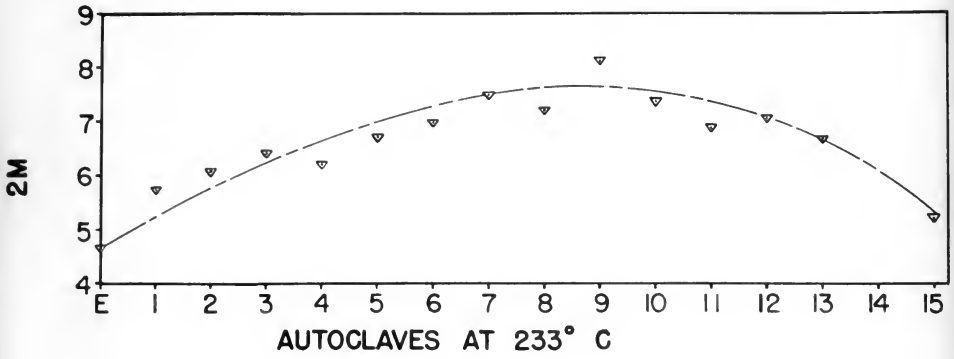
Av ΔR -1.73 PHASE ROTATION

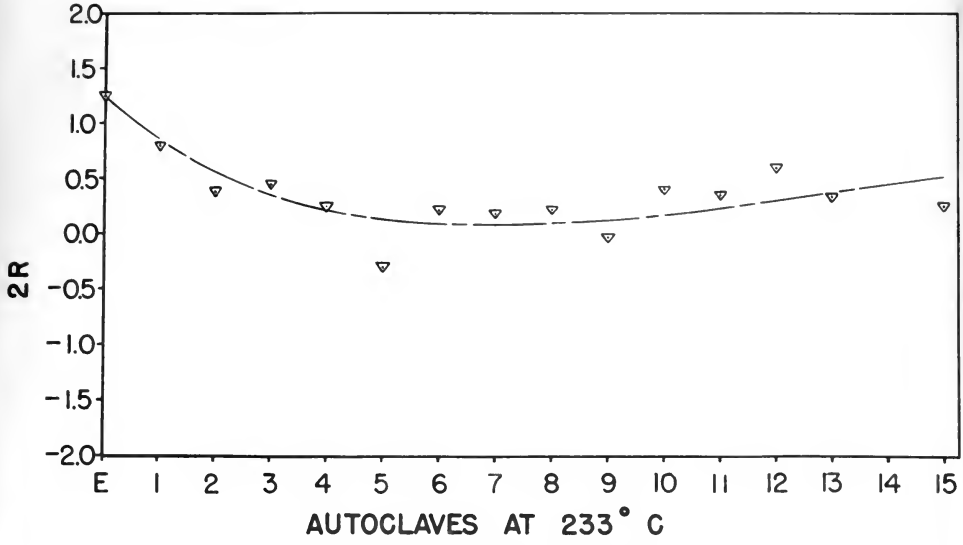
PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





PHASE SHIFT (2m) AND ROTATION OF PLANE OF POLARIZATION (2r) VS. CORROSION TIME FOR SAMPLE NO. 1F-1

SAMPLE No 1F 240 PPM H_2S DATE 6 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION AS ELECTROPOLISHED

	@ 256°		@ 166°		@ 076°		@ 346°	
	M1	R1	M2	R2	M3	R3	M4	R4
	45.1	46.1	40.3	45.0	44.9	46.1	40.5	44.7
	45.3	46.1	40.3	45.0	44.9	46.2	40.5	44.8
Av.	45.20	46.10	40.30	45.00	44.90	46.15	40.5	44.75

M_1	45.20	R_1	46.10
M_2	40.30	R_2	45.00
ΔM_{12}	4.90	ΔR_{12}	1.10

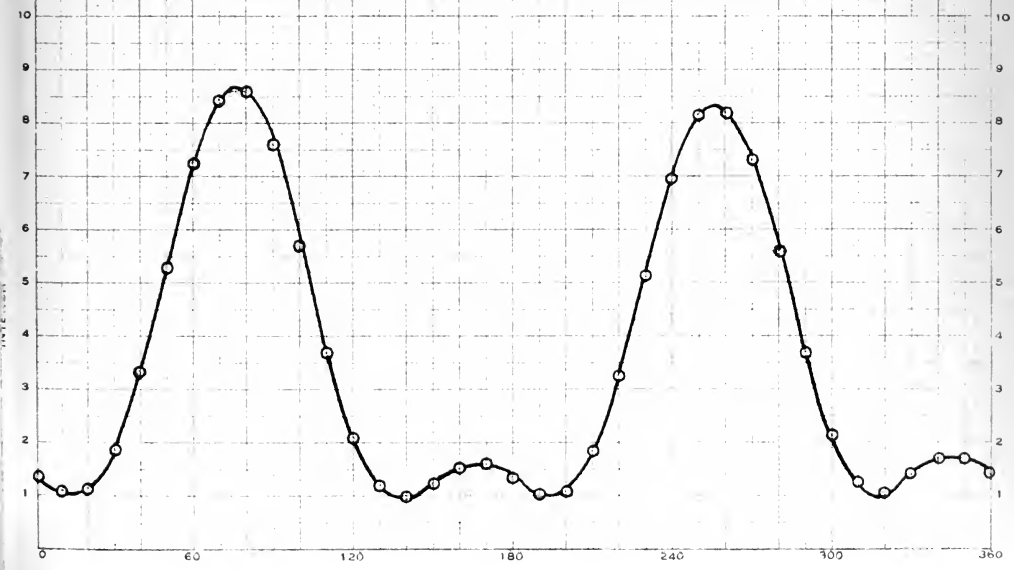
M_3	44.9	R_3	46.15
M_4	40.5	R_4	44.75
ΔM_{34}	4.40	ΔR_{34}	1.40

Av ΔM 4.65 PHASE SHIFT

Av ΔR 1.25 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 1F 240 PPM N_2 DATE 12 MARCH 1952 AM

CRYSTAL No 1 SURFACE FILM CONDITION 1st AUTOCLAVE 15 MIN. at 233°C

	@ 006		@ 276		@ 186		@ 096	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	45.2	45.9	39.2	45.1	45.1	45.9	39.5	45.1
	45.0	45.9	39.4	45.1	45.0	45.9	39.3	45.1
Av.	45.10	45.90	39.30	45.10	45.05	45.90	39.40	45.10

M_1	45.10	R_1	45.90
M_2	39.30	R_2	45.10
ΔM_{12}	5.80	ΔR_{12}	0.80

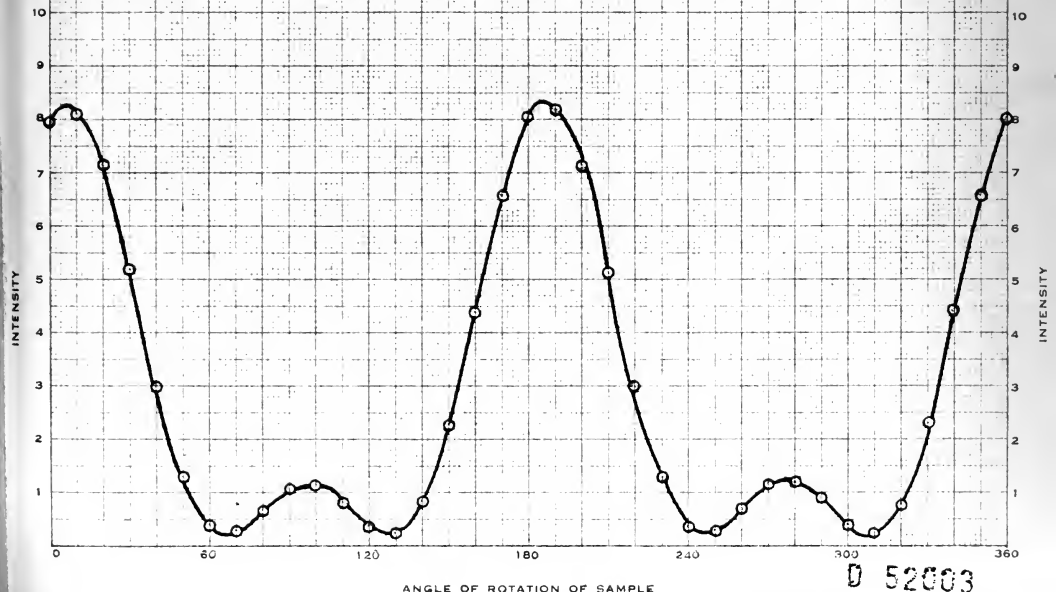
M_3	45.05	R_3	45.90
M_4	39.40	R_4	45.10
ΔM_{34}	5.65	ΔR_{34}	0.80

Av ΔM 5.72 PHASE SHIFT

Av ΔR 0.80 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM N_2 DATE 14 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 2nd AUTOCLAVE 15 MIN. at 233°C

	@ .005		@ .275		@ .185		@ .095	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	46.4	45.5	40.2	45.2	46.2	45.6	40.2	45.2
	46.4	45.4	40.2	45.2	46.1	45.7	40.2	45.2
Av.	46.40	45.50	40.20	45.20	46.15	45.65	40.20	45.20

M_1	46.40	R_1	45.50
M_2	40.20	R_2	45.20
ΔM_{12}	6.20	ΔR_{12}	0.30

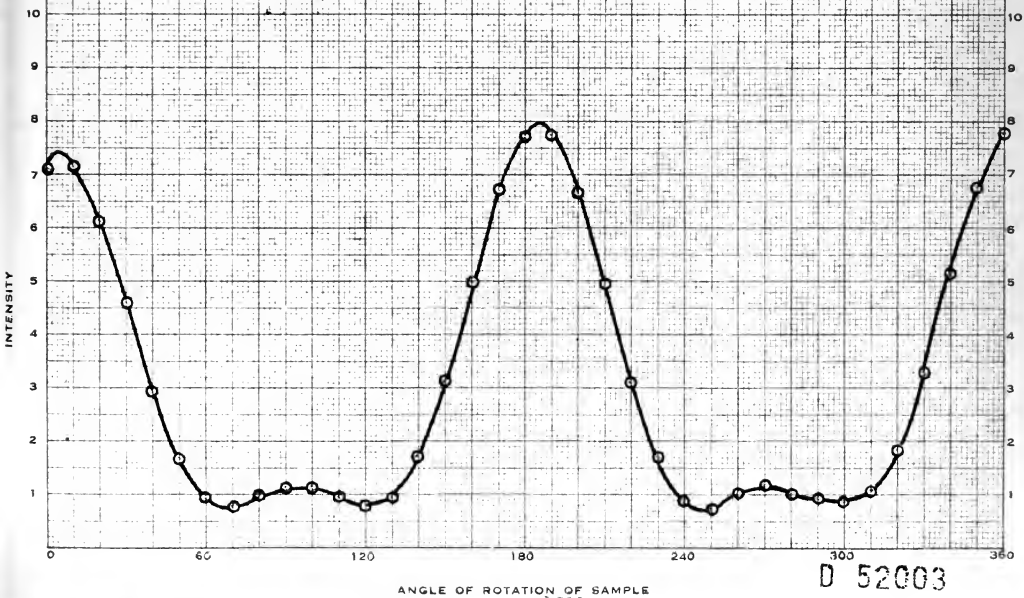
M_3	46.15	R_3	45.65
M_4	40.20	R_4	45.20
ΔM_{34}	5.95	ΔR_{34}	0.45

Av ΔM 6.08 PHASE SHIFT

Av ΔR 0.38 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 242 PPM N_2 DATE 17 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 3rd AUTOCLAVE 15 MIN. at 233°C

	@ 333		@ 243		@ 153		@ 063	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	46.2	45.7	39.9	45.3	46.30	45.80	39.7	45.2
	46.3	45.7	40.1	45.3	46.20	45.70	39.7	45.3
Av.	46.25	45.70	40.00	45.30	46.25	45.75	39.70	45.25

M_1	46.25	R_1	45.70
M_2	40.50	R_2	45.30
ΔM_{12}	6.25	ΔR_{12}	0.40

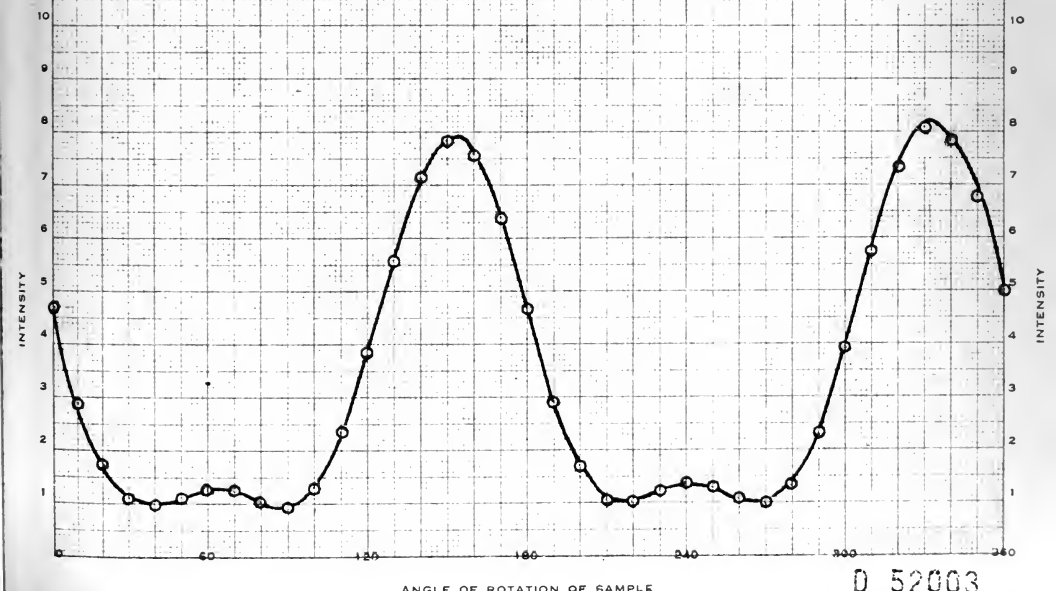
M_3	46.25	R_3	45.75
M_4	39.70	R_4	45.25
ΔM_{34}	6.55	ΔR_{34}	0.50

Av ΔM 6.40 PHASE SHIFT

Av ΔR 0.45 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 11 PPM H_2 DATE 22 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 4th AUTOCLAVE 15 MIN. @ 233°C

	@ 282		@ 192		@ 102		@ 012	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	45.6	45.8	39.6	45.7	45.6	45.9	39.2	45.7
	45.6	46.0	39.6	45.7	45.4	46.1	39.0	45.7
Av.	45.60	45.90	39.60	45.70	45.50	46.00	39.10	45.70

M_1	45.60	R_1	45.90
M_2	39.60	R_2	45.70
ΔM_{12}	6.00	ΔR_{12}	0.20

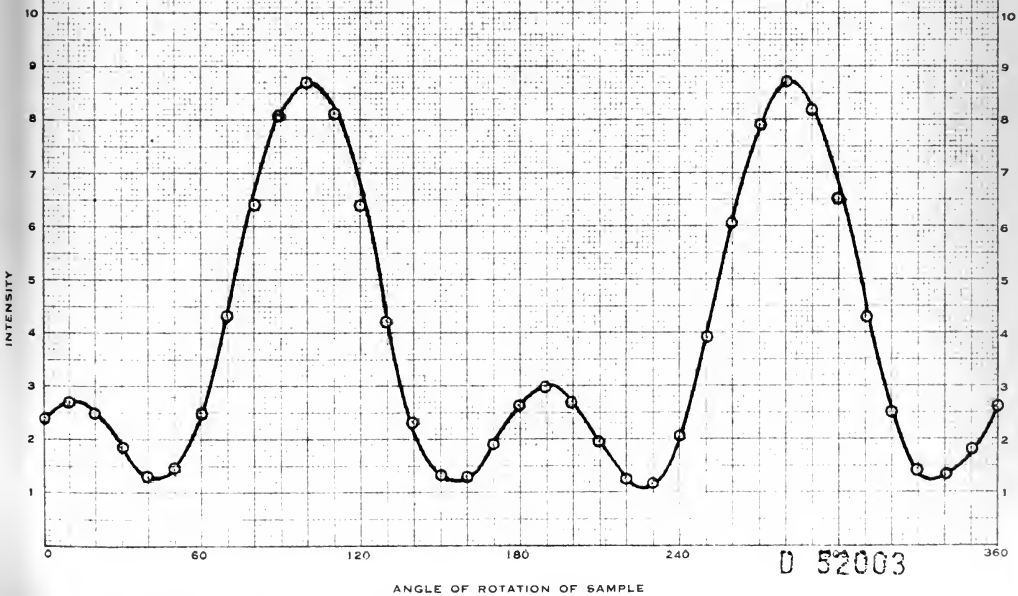
M_3	45.50	R_3	46.00
M_4	39.10	R_4	45.70
ΔM_{34}	6.40	ΔR_{34}	0.30

Av ΔM 6.20 PHASE SHIFT

Av ΔR 0.25 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



SAMPLE No 1F PPM DATE 24 MARCH 1952 PM

CRYSTAL No 1 SURFACE FILM CONDITION 5th AUTOCLAVE 15 MIN @ 232°C

	@ 327		@ 237		@ 147		@ 057	
	M	R1	M2	R2	M3	R3	M4	R4
	45.0	46.0	38.2	45.8	44.7	46.0	38.0	45.7
	45.0	46.0	38.4	45.7	44.7	46.0	38.0	45.6
Av.	45.00	46.00	38.30	45.75	44.70	46.00	38.00	45.65

M_1	45.00	R_1	46.00
M_2	38.30	R_2	45.75
ΔM_{12}	6.70	ΔR_{12}	-0.25

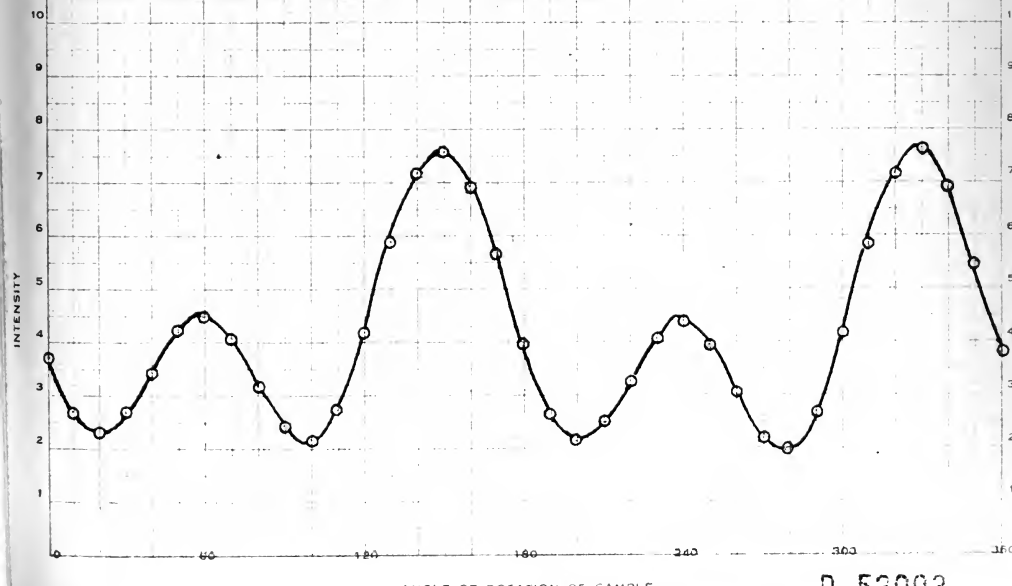
M_3	44.70	R_3	46.00
M_4	38.00	R_4	45.65
ΔM_{34}	6.70	ΔR_{34}	-0.35

Av ΔM 6.70 PHASE SHIFT

Av ΔR -0.30 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 250 PPM 2 DATE 25 MARCH 1952 PM

CRYSTAL No 1 SURFACE FILM CONDITION 6th AUTOCLAVE 15 MIN. @ 233°C

	@ 026		@ 296		@ 206		@ 116	
	M_1	R_1	M_3	R_2	M_3	R_3	M_4	R_4
	47.8	46.0	41.0	45.6	47.7	45.9	40.4	45.7
	47.7	46.0	41.0	45.7	47.5	45.8	40.4	45.8
Av.	47.75	46.00	41.00	45.65	47.60	45.85	40.40	45.75

M_1	47.75	R_1	46.00
M_2	41.00	R_2	45.65
ΔM_{12}	6.75	ΔR_{12}	0.35

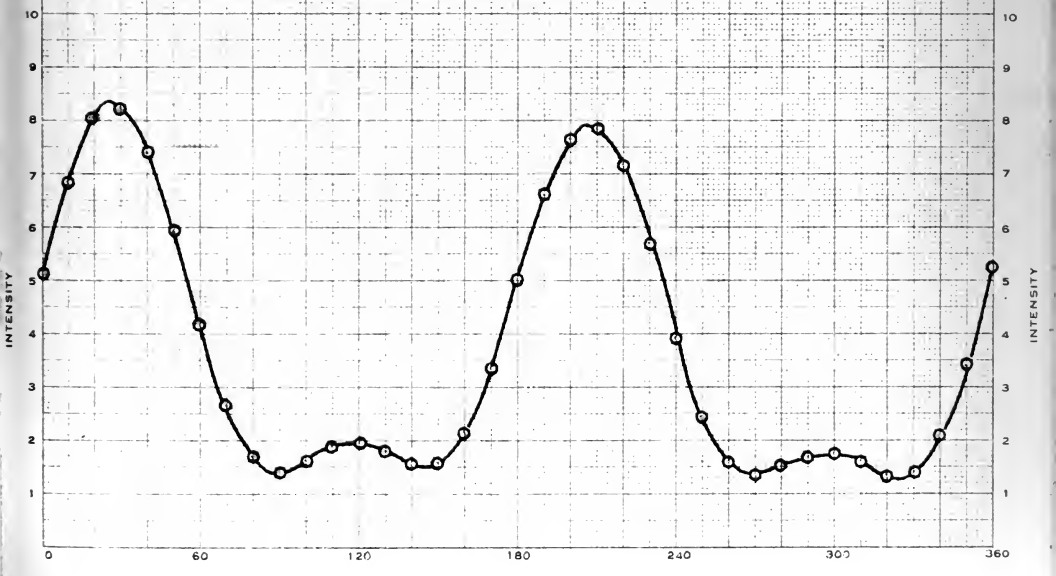
M_3	47.60	R_3	45.85
M_4	40.40	R_4	45.75
ΔM_{34}	7.20	ΔR_{34}	0.10

Av ΔM 6.97 PHASE SHIFT

Av ΔR 0.22 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM N_2 DATE 26 MARCH 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 7th AUTOCLAVE 15 MIN. @ 233°C

	@ 315		@ 225		@ 135		@ 045	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	47.3	45.5	39.8	45.4	47.1	45.6	39.7	45.4
	47.4	45.6	39.7	45.4	47.1	45.6	39.8	45.4
Av.	47.35	45.55	39.75	45.40	47.10	45.6	39.75	45.40

M_1	47.35	R_1	45.56
M_2	39.75	R_2	45.40
ΔM_{12}	7.60	ΔR_{12}	0.15

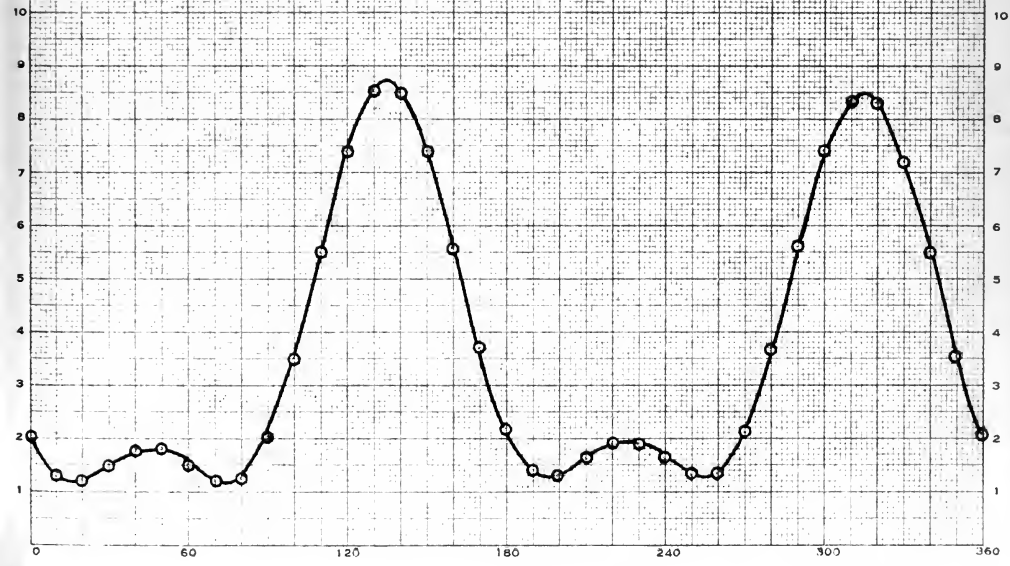
M_3	47.10	R_3	45.60
M_4	39.75	R_4	45.40
ΔM_{34}	7.35	ΔR_{31}	0.20

Av ΔM 7.48 PHASE SHIFT

Av ΔR 0.18 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 1F 240 PPM H_2 DATE 27 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 8th AUTOCLAVE 15 MIN. at 238°C

	@ 039		@ 309		@ 219		@ 129	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	46.9	45.5	39.7	45.3	46.8	45.6	39.7	45.3
	47.0	45.5	39.6	45.4	46.7	45.6	39.6	45.3
Av.	46.95	45.50	39.65	45.35	46.75	45.60	39.65	45.30

M_1	46.95	R_1	45.50
M_2	39.65	R_2	45.35
ΔM_{12}	7.30	ΔR_{12}	0.15

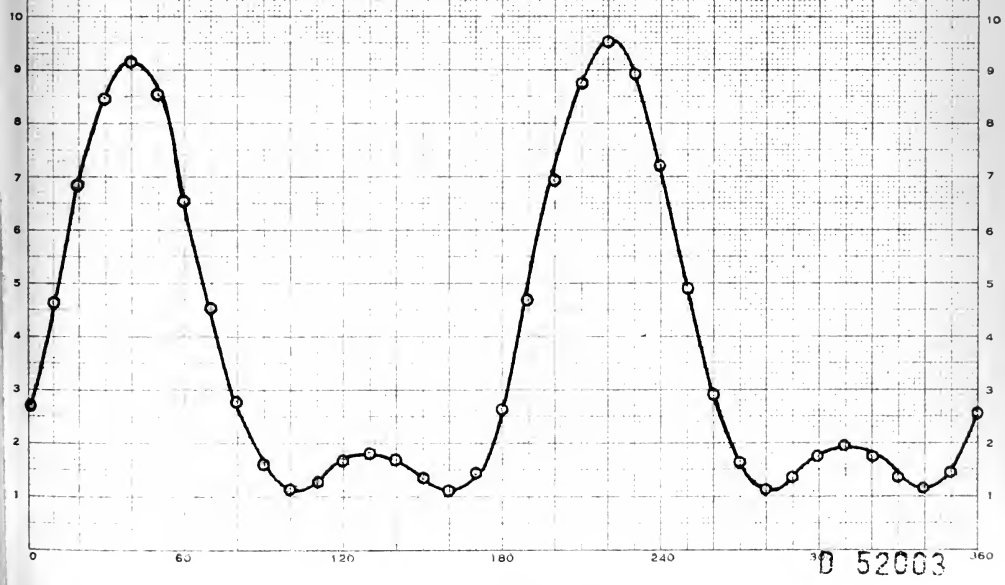
M_3	46.75	R_3	45.60
M_4	39.65	R_4	45.30
ΔM_{34}	7.10	ΔR_{34}	0.30

Av ΔM 7.20 PHASE SHIFT

Av ΔR 0.22 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM N_2 DATE 28 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 9th AUTOCLAVE 15 MIN @ 233°C

	@ 028		@ 298		@ 208		@ 118	
	M	R	M_2	R_2	M_3	R_3	M_4	R_4
	48.1	45.6	39.6	45.1	47.7	45.6	39.8	45.4
	47.8	45.4	39.1	45.7	47.7	45.5	39.7	45.4
Av.	47.95	45.50	39.65	45.70	47.70	45.55	39.75	45.40

M_1	47.95	R_1	45.50
M_2	39.65	R_2	45.70
ΔM_{12}	8.30	ΔR_{12}	-0.20

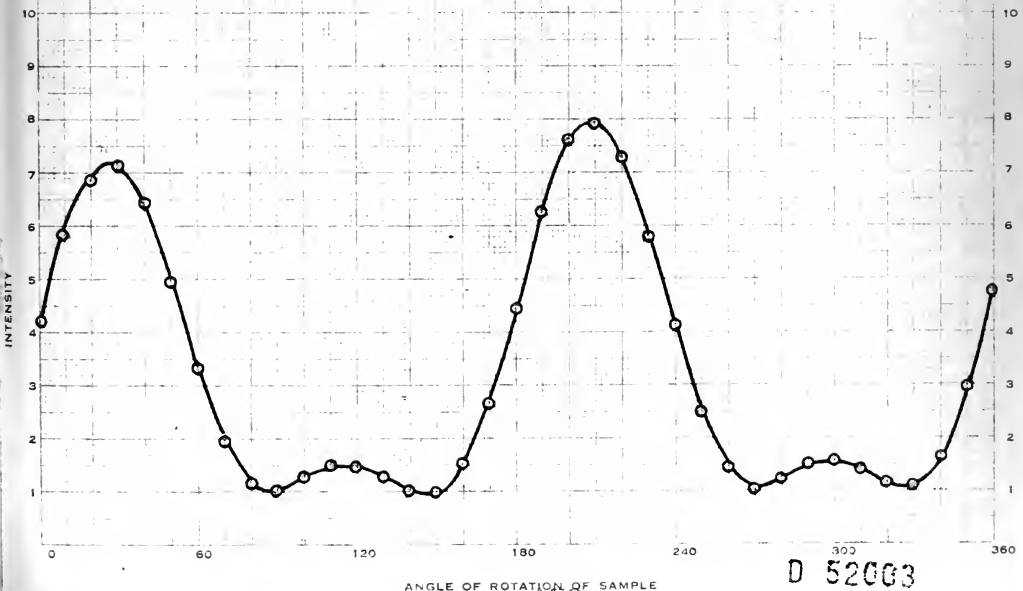
M_3	47.70	R_3	45.55
M_4	39.75	R_4	45.40
ΔM_{34}	7.95	ΔR_{34}	0.15

Av ΔM 8.12 PHASE SHIFT

Av ΔR -0.03 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM N_2 DATE 31 MARCH 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 10th AUTOCLAVE 15 MIN. @ 233°C

	@ 225		@ 135		@ 045		@ 315	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	47.7	46.0	40.5	45.5	47.5	45.9	40.0	45.6
	47.7	46.1	40.5	45.7	47.6	45.8	40.0	45.4
Av.	47.70	46.05	40.50	45.60	47.55	45.85	40.00	45.50

M_1	47.70	R_1	46.05
M_2	40.50	R_2	45.60
ΔM_{12}	7.20	ΔR_{12}	0.45

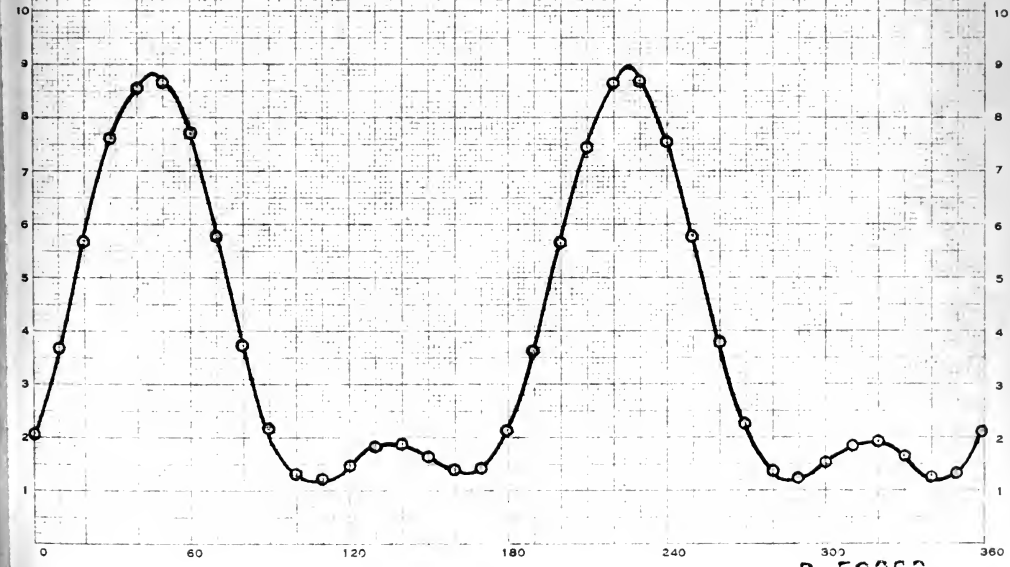
M_3	47.55	R_3	45.85
M_4	40.00	R_4	45.50
ΔM_{34}	7.55	ΔR_{34}	0.35

Av ΔM 7.37 PHASE SHIFT

Av ΔR 0.40 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM N_2 DATE 1 APRIL 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 11th AUTOCLAVE 15 MIN. @ 233°C

	@ 326		@ 236		@ 146		@ 056	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	45.5	45.6	38.7	45.4	45.5	45.8	38.5	45.4
	45.3	45.7	38.6	45.3	45.5	45.7	38.5	45.3
Av.	45.40	45.65	38.65	45.35	45.50	45.75	38.50	45.35

M_1	45.40	R_1	45.65
M_2	38.65	R_2	45.35
ΔM_{12}	6.75	ΔR_{12}	0.30

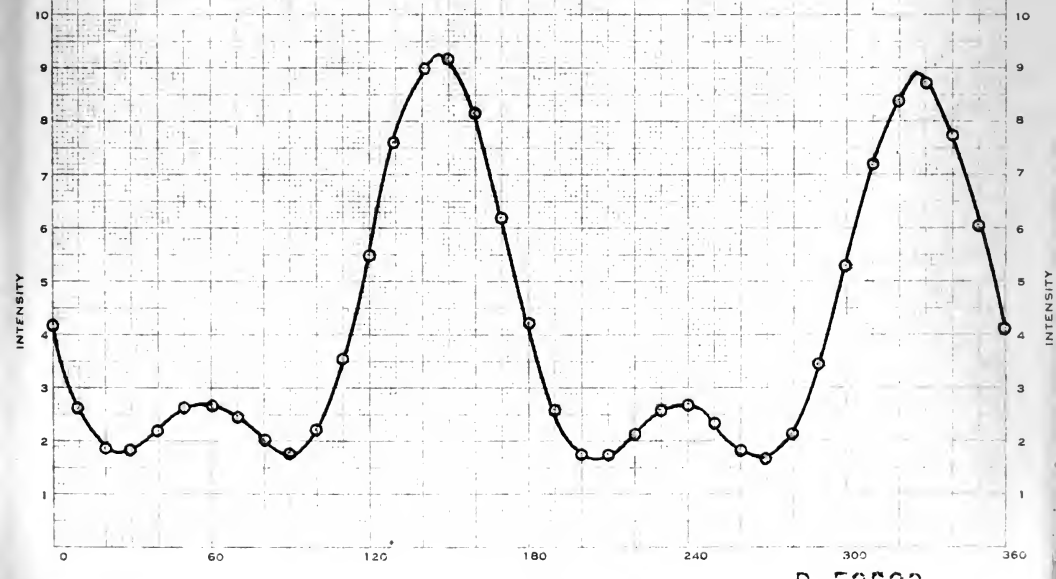
M_3	45.50	R_3	45.75
M_4	38.50	R_4	45.35
ΔM_{34}	7.00	ΔR_{34}	0.40

Av ΔM 6.87 PHASE SHIFT

Av ΔR 0.35 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM N_2 DATE 2 APRIL 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 12th AUTOCLAVE 15 MIN @ 233°C

	@ 222		@ 132		@ 042		@ 312	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	45.7	45.9	38.3	45.1	45.4	45.5	38.7	45.2
	45.4	45.7	38.0	45.0	45.4	45.7	32.8	45.1
Av.	45.65	45.80	38.15	45.05	45.40	45.60	38.80	45.15

M_1	45.65	R_1	45.80
M_2	38.15	R_2	45.05
ΔM_{12}	7.50	ΔR_{12}	.75

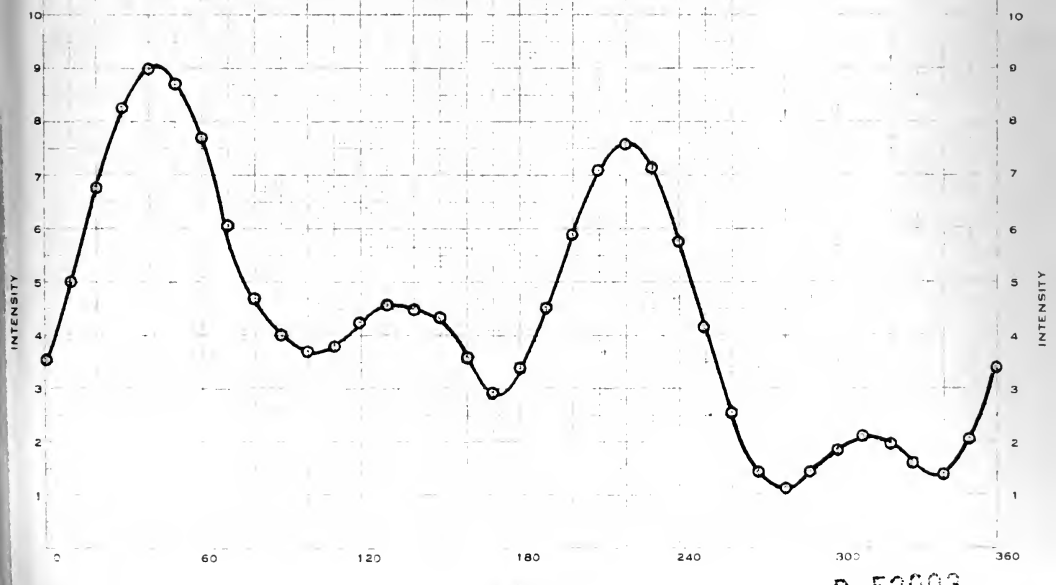
M_3	45.40	R_3	45.60
M_4	38.80	R_4	45.15
ΔM_{34}	6.60	ΔR_{34}	.45

Av ΔM 7.05 PHASE SHIFT

Av ΔR 0.60 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM N_2 DATE 4 APRIL 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 13th AUTOCLAVE 15 MIN. @ 233°C

	@ 210		@ 120		@ 030		@ 300	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	45.3	46.1	38.9	45.8	45.5	46.0	38.6	45.7
	45.5	46.0	38.9	45.6	45.5	46.0	38.7	45.7
Av.	45.40	46.05	38.90	45.70	45.50	46.00	38.65	45.70

M_1	45.40	R_1	46.05
M_2	38.90	R_2	45.70
ΔM_{12}	6.50	ΔR_{12}	0.35

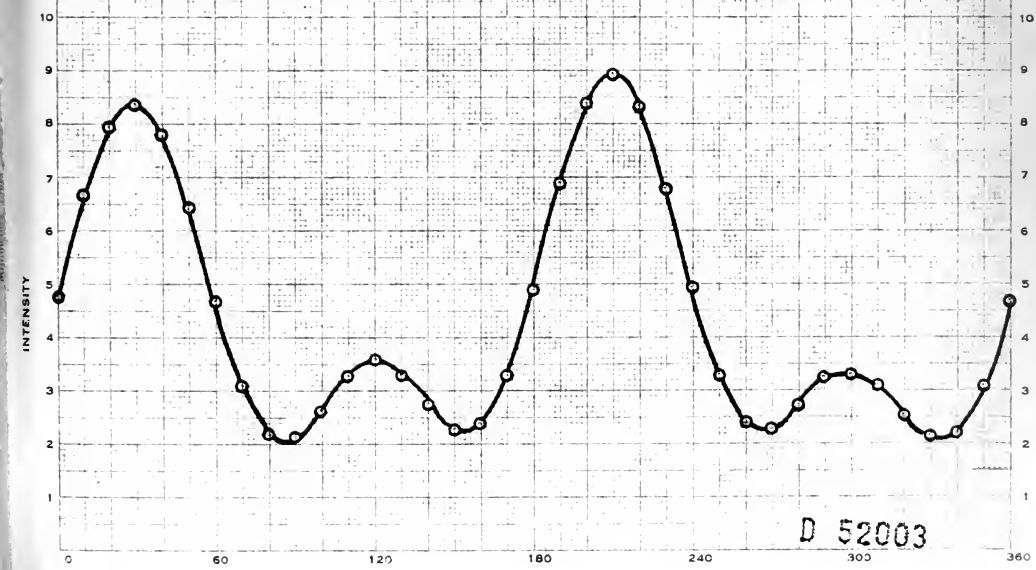
M_3	45.50	R_3	46.00
M_4	38.65	R_4	45.70
ΔM_{34}	6.85	ΔR_{34}	0.30

Av ΔM 6.67 PHASE SHIFT

Av ΔR 0.33 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



SAMPLE No. 1F 240 PPM N_2 DATE 5 APRIL 1952 AM

CRYSTAL No. 1 SURFACE FILM CONDITION 15th AUTOCLAVE OF 15 MIN. @ 233°C

	@ 264		@ 174		@ 084		@ 354	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	47.2	45.8	41.9	45.8	46.8	46.0	41.6	45.8
	46.9	46.0	41.8	45.7	46.8	46.0	41.6	45.7
Av.	47.05	45.90	41.85	45.75	46.80	46.00	41.60	45.75

M_1	47.05	R_1	45.90
M_2	41.85	R_2	45.75
ΔM_{12}	5.20	ΔR_{13}	0.25

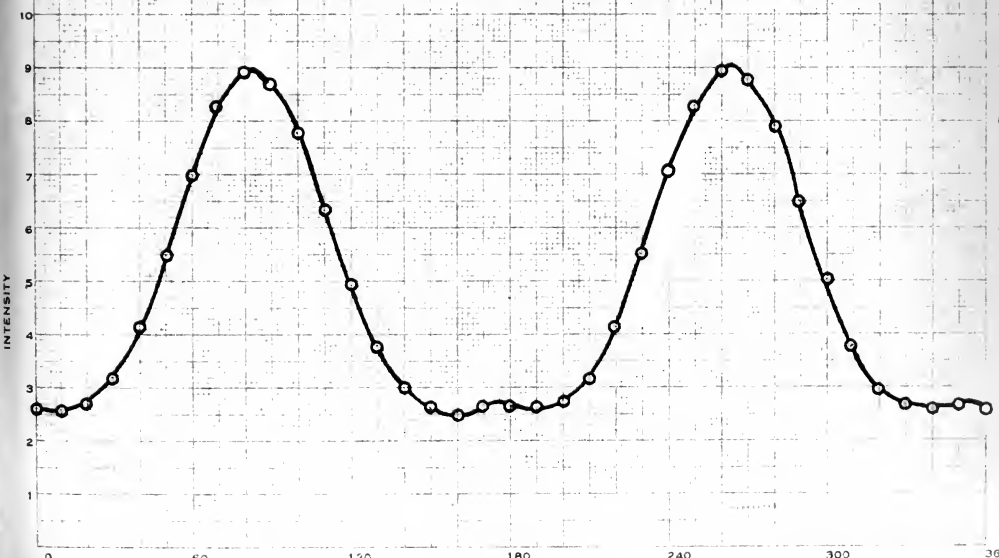
M_3	46.80	R_3	46.00
M_4	41.50	R_4	45.75
ΔM_{34}	5.20	ΔR_{24}	0.25

Av ΔM 5.20 PHASE SHIFT

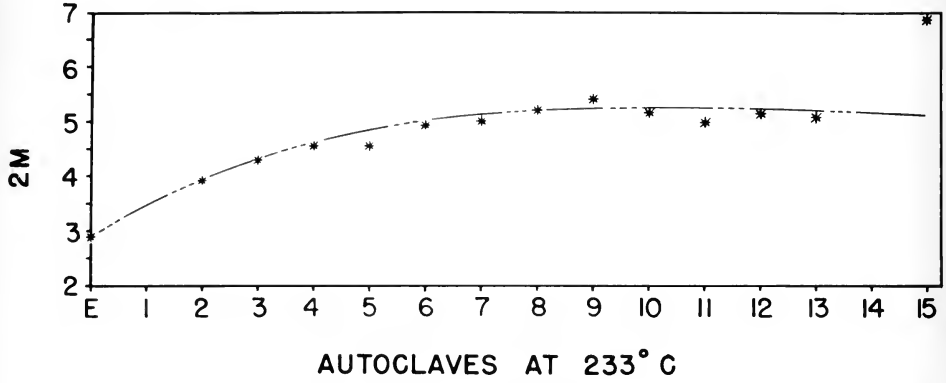
Av ΔR 0.25 PHASE ROTATION

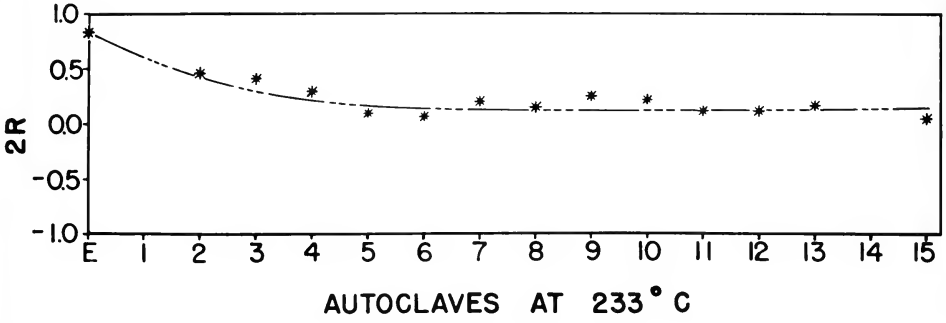
PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





PHASE SHIFT (2m) AND ROTATION OF PLANE OF POLARIZATION (2ρ) VS. CORROSION TIME FOR SAMPLE NO. 1F-2

D 52083

SAMPLE No. 1F 240 PPM N_2 DATE 6 MARCH 1952 AM

CRYSTAL No. 2 SURFACE FILM CONDITION AS ELECTROPOLISHED

	@ 344°		@ 254°		@ 164°		@ 074°	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	44.2	45.8	41.2	45.2	43.9	46.2	41.2	45.2
	44.2	46.0	41.1	45.1	44.0	45.9	41.2	45.1
Av.	44.20	45.90	41.15	45.15	43.95	46.05	41.20	45.15

M_1	44.20	R_1	45.90
M_2	41.15	R_2	45.15
ΔM_{12}	3.05	ΔR_{12}	0.75

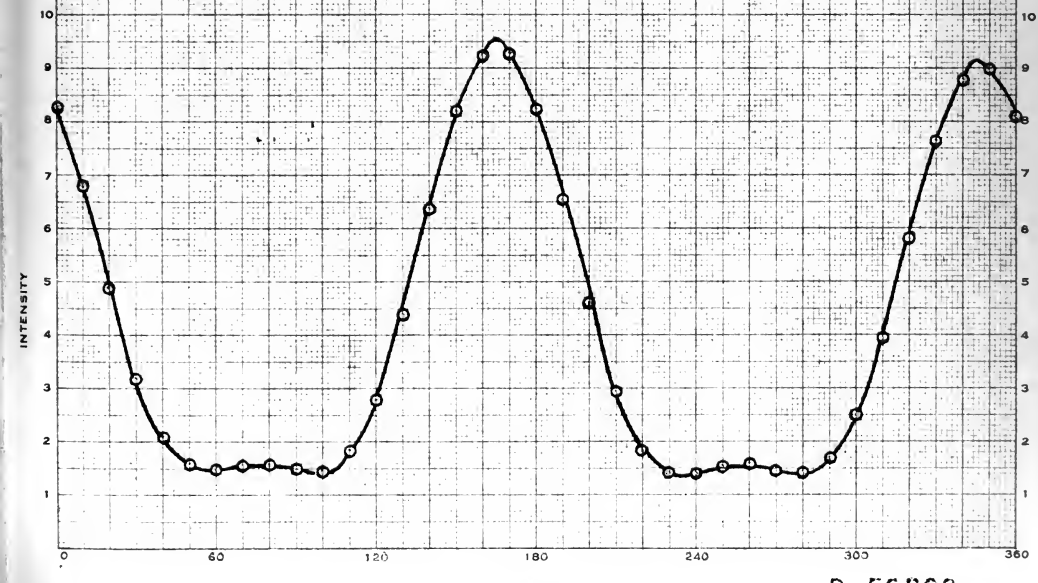
M_3	43.95	R_3	46.05
M_4	41.20	R_4	45.15
ΔM_{34}	2.75	ΔR_{34}	0.90

Av ΔM 2.90 PHASE SHIFT

Av ΔR 0.83 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 12 240 PPM N_2 DATE 12 MARCH 1952 AM

CRYSTAL No. 2 SURFACE FILM CONDITION 1st AUTOCLAVE 15 MIN. 6 23° C

	@ 276		@ 186		@ 096		@ 006	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	44.1	45.8	40.0	45.2	43.7	45.8	40.2	45.3
	43.7	45.6	40.1	45.3	43.7	45.8	40.2	45.4
	43.9	45.8						
Av.	43.90	45.73	40.05	45.25	43.70	45.8	40.2	45.35

M_1	43.90	R_1	45.73
M_2	40.05	R_2	45.25
ΔM_{12}	3.85	ΔR_{12}	0.48

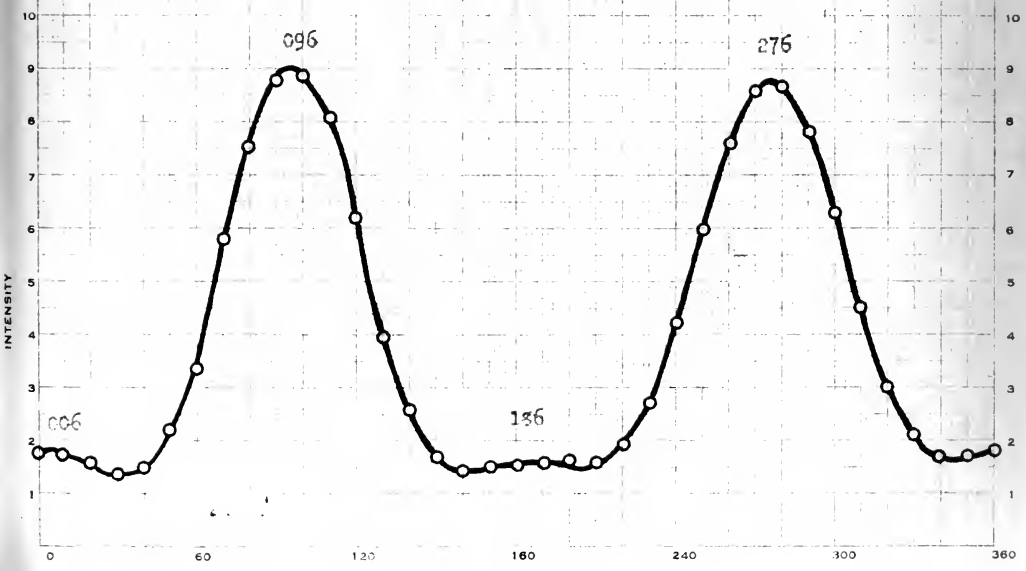
M_3	43.70	R_3	45.80
M_4	40.20	R_4	45.35
ΔM_{34}	3.50	ΔR_{34}	0.45

Av ΔM 3.67 PHASE SHIFT

Av ΔR 0.46 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM N_2 DATE 14 MARCH 1952

CRYSTAL No. 2 SURFACE FILM CONDITION 2nd AUTOCLAVE 15 MIN. at 233°C

	@ 275		@ 185		@ 095		@ 005	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	45.2	45.8	41.2	45.3	45.1	45.7	41.2	45.2
	45.2	45.7	41.4	45.3	45.1	45.7	41.1	45.2
Av.	45.2	45.75	41.30	45.30	45.10	45.70	41.15	45.20

M_1	45.20	R_1	45.75
M_2	41.30	R_2	45.30
ΔM_{12}	3.90	ΔR_{12}	0.45

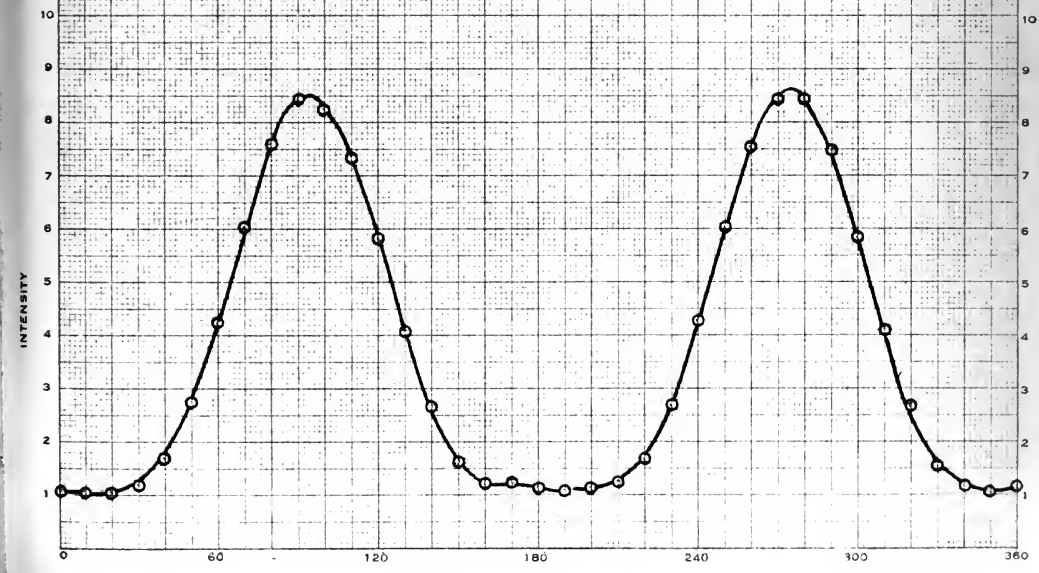
M_3	45.10	R_3	45.70
M_4	41.15	R_4	45.20
ΔM_{34}	3.95	ΔR_{34}	0.50

Av ΔM 3.92 PHASE SHIFT

Av ΔR 0.47 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. ___ PPM 2 DATE ___

CRYSTAL No. 2 SURFACE FILM CONDITION 3rd AUTOCLAVE 15 MIN. at 233°C

	@ 242		@ 152		@ 062		@ 332	
	M1	R1	M2	R2	M3	R3	M4	R4
	45.1	45.8	41.0	45.30	45.0	45.8	40.7	45.4
	45.2	45.8	40.8	45.40	45.1	45.8	40.7	45.4
Av.	45.15	45.30	40.90	45.35	45.05	45.80	40.70	45.40

M_1	45.15	R_1	45.80
M_2	40.30	R_2	45.35
ΔM_{12}	4.25	ΔR_{12}	0.45

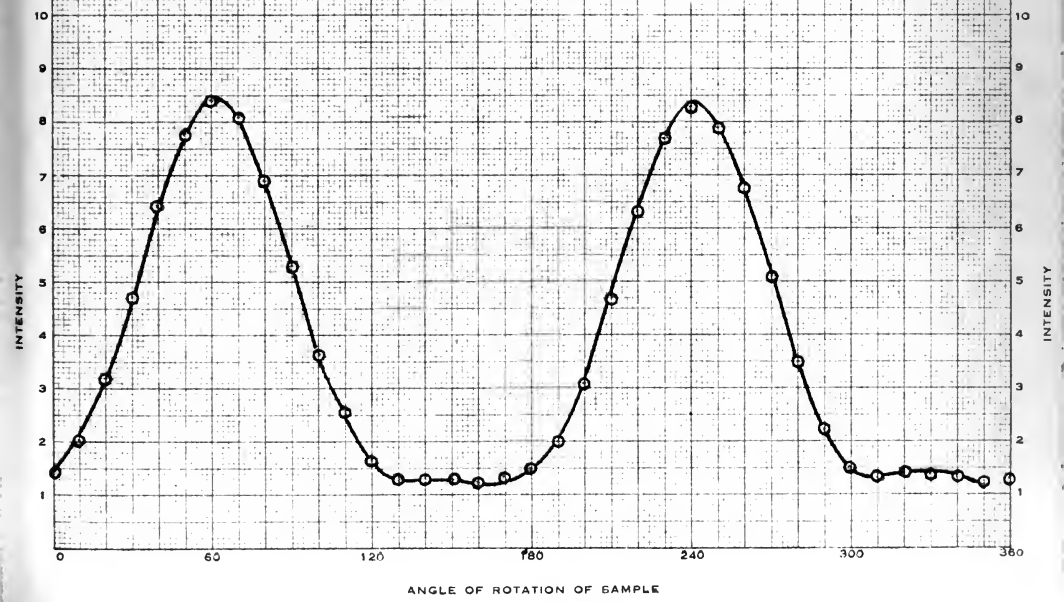
M_3	45.05	R_3	45.80
M_4	40.70	R_4	45.40
ΔM_{34}	4.35	ΔR_{34}	0.40

Av ΔM 4.30 PHASE SHIFT

Av ΔR 0.42 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



SAMPLE No. [illegible] DATE [illegible]

CRYSTAL No. 2 SURFACE FILM CONDITION 4th AUTOCLAVE 15 MIN. @ 233°C

	@ 011		@ 281		@ 191		@ 101	
	M1	R1	M2	R2	M3	R3	M4	R4
	44.6	46.1	40.1	45.7	44.5	45.9	40.0	45.7
	44.6	46.1	40.0	45.7	44.4	45.9	39.8	45.7
Av.	44.60	46.10	40.05	45.70	44.45	45.90	39.90	45.70

M_1	44.60	R_1	46.10
M_2	40.05	R_2	45.70
ΔM_{12}	4.55	ΔR_{12}	0.40

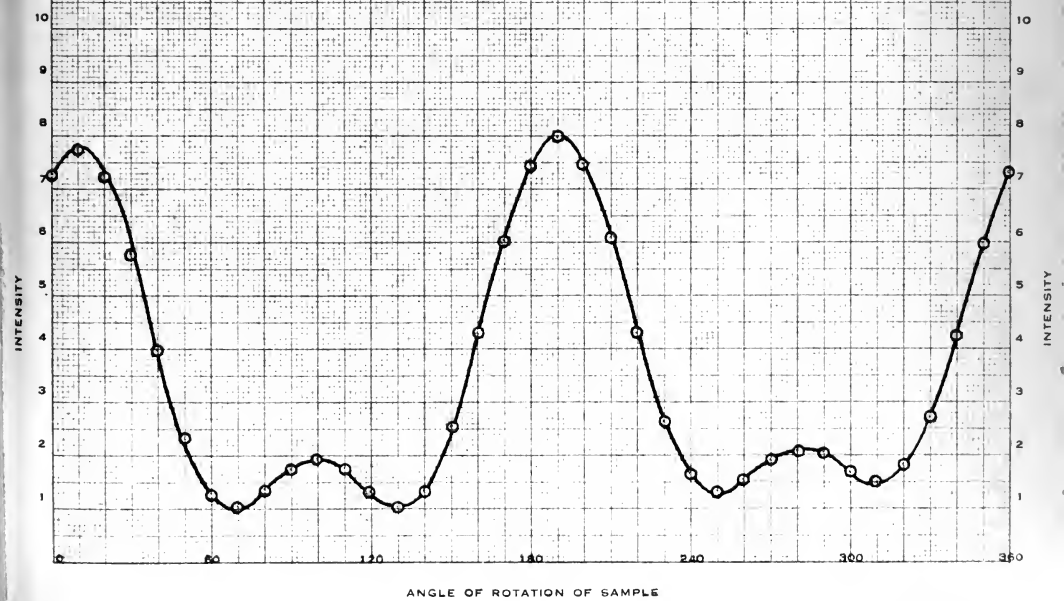
M_3	44.45	R_3	45.90
M_4	39.90	R_4	45.70
ΔM_{34}	4.55	ΔR_{34}	0.20

Av ΔM 4.55 PHASE SHIFT

Av ΔR 0.30 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

CRYSTAL No. 2 SURFACE FILM CONDITION 5th AUTOCLAVE 15 MIN. @ 233°C

	@ 237		@ 147		@ 057		@ 327	
	M1	R1	M2	R2	M3	R3	M4	R4
	43.3	45.7	38.8	45.4	43.4	45.6	38.7	45.7
	43.4	45.6	38.9	45.6	43.2	45.7	38.7	45.6
Av	43.35	45.65	38.85	45.50	43.30	45.65	38.70	45.60

M_1	43.35	R_1	45.65
M_2	38.85	R_2	45.50
ΔM_{12}	4.50	ΔR_{12}	0.15

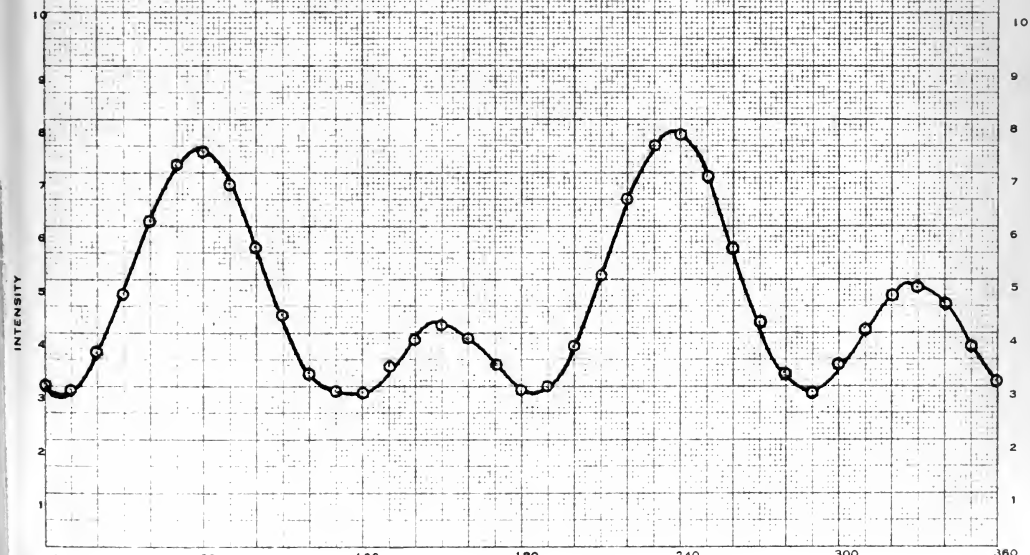
M_3	43.30	R_3	45.65
M_4	38.70	R_4	45.60
ΔM_{34}	4.60	ΔR_{34}	0.05

Av ΔM 4.55 PHASE SHIFT

Av ΔR 0.10 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM Mg^{++} DATE 25 MARCH 1952 PM

CRYSTAL No. 2 SURFACE FILM CONDITION 6th AUTOCLAVE 15 MIN. @ 233°C

	@ 295		@ 205		@ 115		@ 025	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	46.0	45.6	40.8	45.4	45.1	45.7	40.6	45.6
	46.2	45.5	40.9	45.5	45.1	45.6	40.5	45.6
Av.	46.10	45.55	40.85	45.45	45.10	45.65	40.55	45.60

M_1	46.10	R_1	45.55
M_2	40.85	R_2	45.45
ΔM_{12}	5.25	ΔR_{12}	0.10

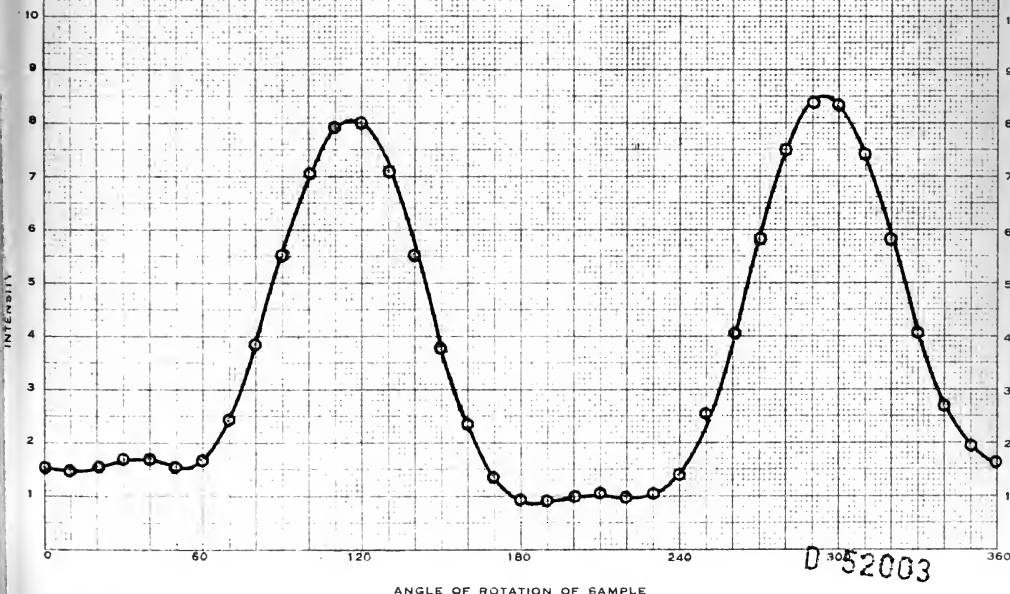
M_3	45.10	R_3	45.65
M_4	40.55	R_4	45.60
ΔM_{34}	4.55	ΔR_{34}	0.05

Av ΔM 4.92 PHASE SHIFT

Av ΔR 0.07 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 40 PPM N_2 DATE 7th MARCH 1952 PM

CRYSTAL No. 2 SURFACE FILM CONDITION 7th AUTOCLAVE 15 MIN. @ 233°C

	@ 224		@ 134		@ 044		@ 314	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	45.5	45.6	40.9	45.4	45.7	45.6	40.5	45.5
	45.7	45.6	40.7	45.4	45.7	45.6	40.5	45.3
Av.	45.60	45.60	40.80	45.40	45.70	45.60	40.50	45.40

M_1	45.60	R_1	45.60
M_2	40.80	R_2	45.40
ΔM_{12}	4.80	ΔR_{12}	0.20

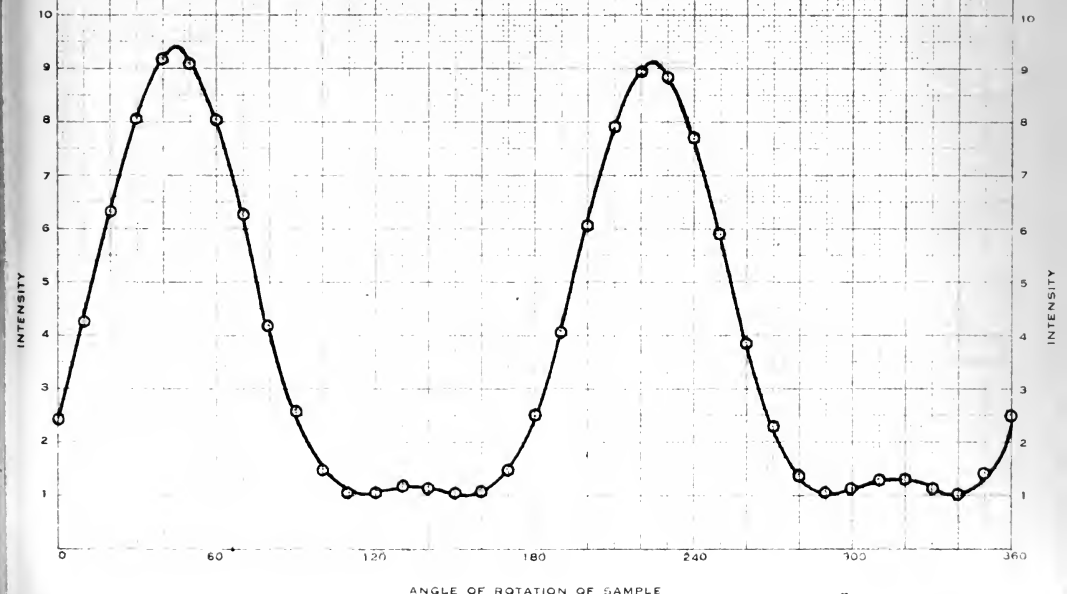
M_3	45.70	R_3	45.60
M_4	40.50	R_4	45.40
ΔM_{34}	5.20	ΔR_{34}	0.20

Av ΔM 5.00 PHASE SHIFT

Av ΔR 0.20 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F [illegible] PPM Cl DATE 27 MARCH 1972 AM

CRYSTAL No. 2 SURFACE FILM CONDITION 8th AUTOCLAVE 15 MIN. @ 235°C

	@ 309		@ 219		@ 129		@ 039	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	46.2	45.6	41.4	45.4	46.4	45.6	40.9	45.6
	46.4	45.7	41.4	45.4	46.2	45.6	40.7	45.5
Av.	46.30	45.65	41.40	45.40	46.30	45.60	40.80	45.55

M_1	46.30	R_1	45.65
M_2	41.40	R_2	45.40
ΔM_{12}	4.90	ΔR_{12}	0.25

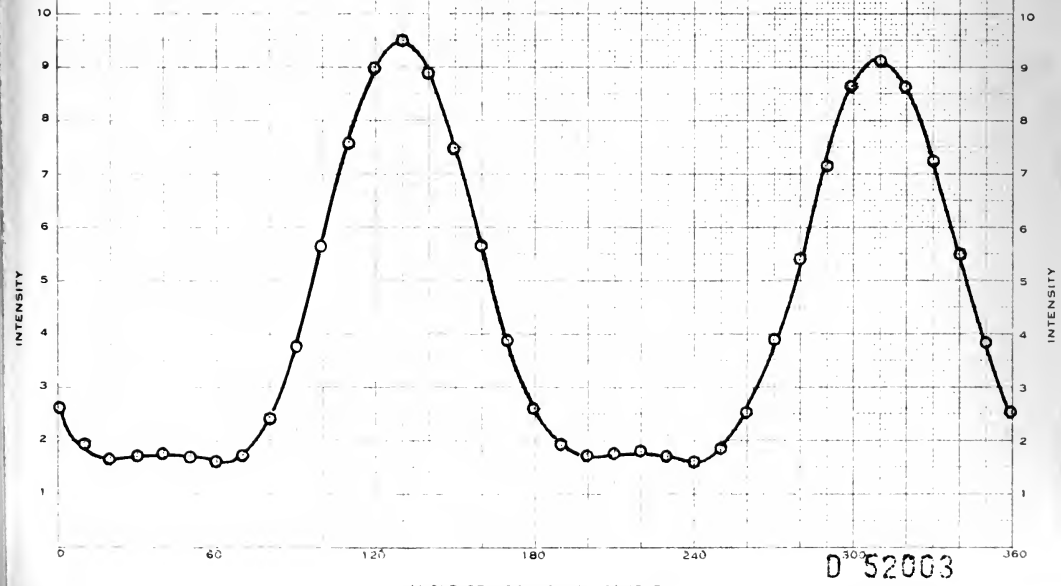
M_3	46.30	R_3	45.60
M_4	40.80	R_4	45.55
ΔM_{34}	5.50	ΔR_{34}	0.05

Av ΔM 5.20 PHASE SHIFT

Av ΔR 0.15 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM H_2 DATE 28 MARCH 1952 AM

CRYSTAL No. 2 , SURFACE FILM CONDITION 9th AUTOCLAVE 15 MIN @ 233°C

	@ 298°		@ 208°		@ 118°		@ 028°	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	47.2	45.7	41.6	45.5	46.9	45.7	41.5	45.3
	47.0	45.7	41.7	45.5	46.8	45.6	41.5	45.4
Av.	47.10	45.70	41.65	45.50	46.85	45.65	41.50	45.35

M_1	47.10	R_1	45.70
M_2	41.65	R_2	45.50
ΔM_{12}	5.45	ΔR_{12}	0.20

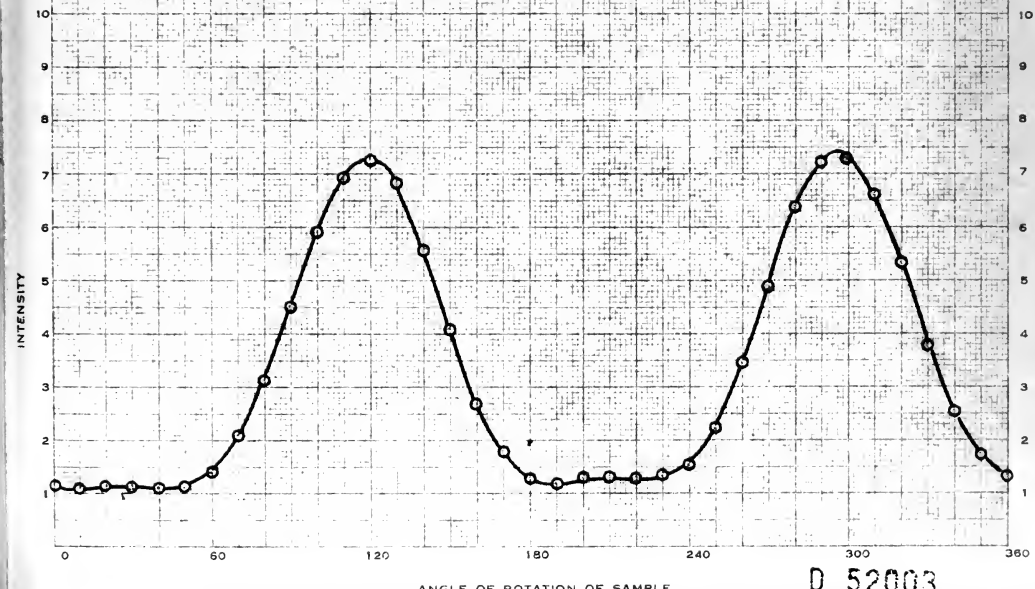
M_3	46.85	R_3	45.65
M_4	41.50	R_4	45.35
ΔM_{34}	5.35	ΔR_{34}	0.30

Av ΔM 5.40 PHASE SHIFT

Av ΔR 0.25 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM H_2 - DATE 31 MARCH 1952 AM

CRYSTAL No. 2 SURFACE FILM CONDITION 10th AUTOCLAVE 15 MIN. @ 233°C

	@ 314		@ 224		@ 134		@ 044	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	46.9	45.8	41.6	45.4	46.6	45.7	41.6	45.7
	46.7	45.8	41.7	45.6	46.9	45.8	41.6	45.5
Av.	46.80	45.80	41.65	45.50	46.75	45.75	41.60	45.60

M_1	46.80	R_1	45.80
M_2	41.65	R_2	45.50
ΔM_{12}	5.15	ΔR_{12}	0.30

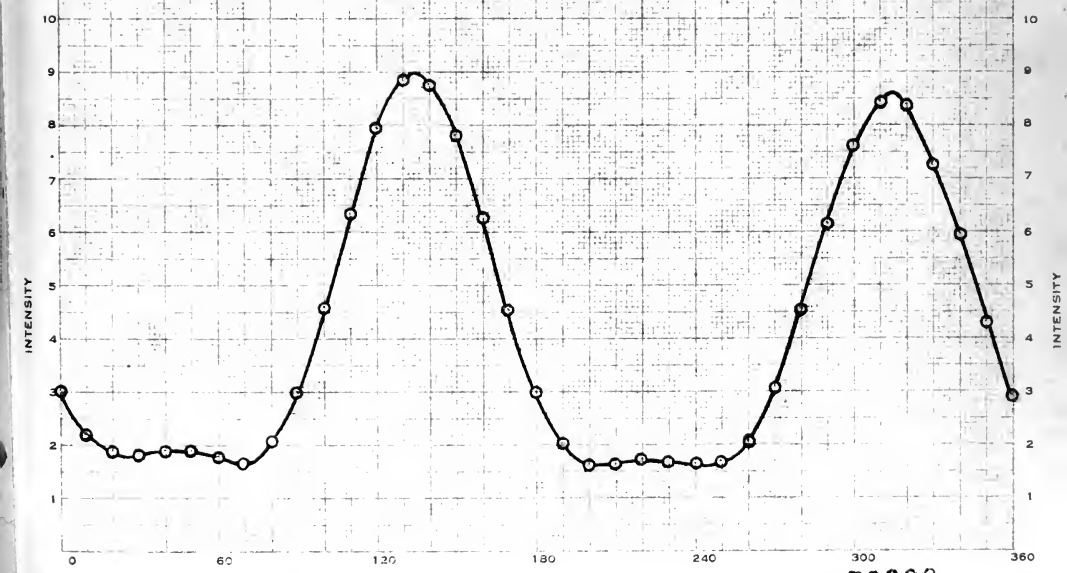
M_3	46.75	R_3	45.75
M_4	41.60	R_4	45.60
ΔM_{34}	5.15	ΔR_{34}	0.15

Av ΔM 5.15 PHASE SHIFT

Av ΔR 0.22 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM H_2 DATE 1 APRIL 1952 AM

CRYSTAL No. 2 SURFACE FILM CONDITION 11th AUTOCLAVE 15 MIN @ 233°C

@ 239		@ 146		@ 056		@ 326	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
44.5	45.8	39.9	45.7	44.8	45.9	39.6	45.6
44.6	45.8	39.6	45.7	44.7	45.7	39.6	45.7
Av. 44.55	45.80	39.75	45.70	44.75	45.80	39.60	45.65

M_1	44.55	R_1	45.80
M_2	39.75	R_2	45.70
ΔM_{12}	4.80	ΔR_{12}	0.10

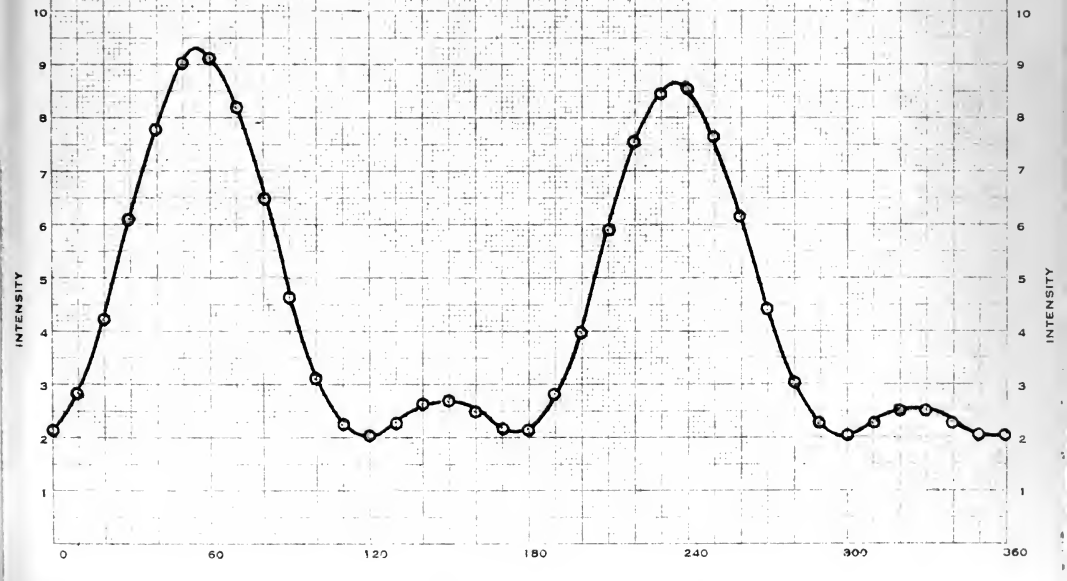
M_3	44.75	R_3	45.80
M_4	39.60	R_4	45.65
ΔM_{34}	5.15	ΔR_{34}	0.15

Av ΔM 4.97 PHASE SHIFT

Av ΔR 0.12 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM N_2 DATE 4 APRIL 1952 AM

CRYSTAL No. 2 SURFACE FILM CONDITION 13th AUTOCLAVE 15 MIN. @ 233°C

	@ 300		@ 210		@ 120		@ 030	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	44.4	45.9	39.8	45.7	45.0	45.9	39.4	45.7
	44.6	45.8	39.6	45.7	44.7	45.7	39.4	45.5
Av.	44.50	45.85	39.70	45.70	44.85	45.80	39.40	45.60

M_1	44.50	R_1	45.85
M_2	39.70	R_2	45.70
ΔM_{12}	4.80	ΔR_{12}	0.15

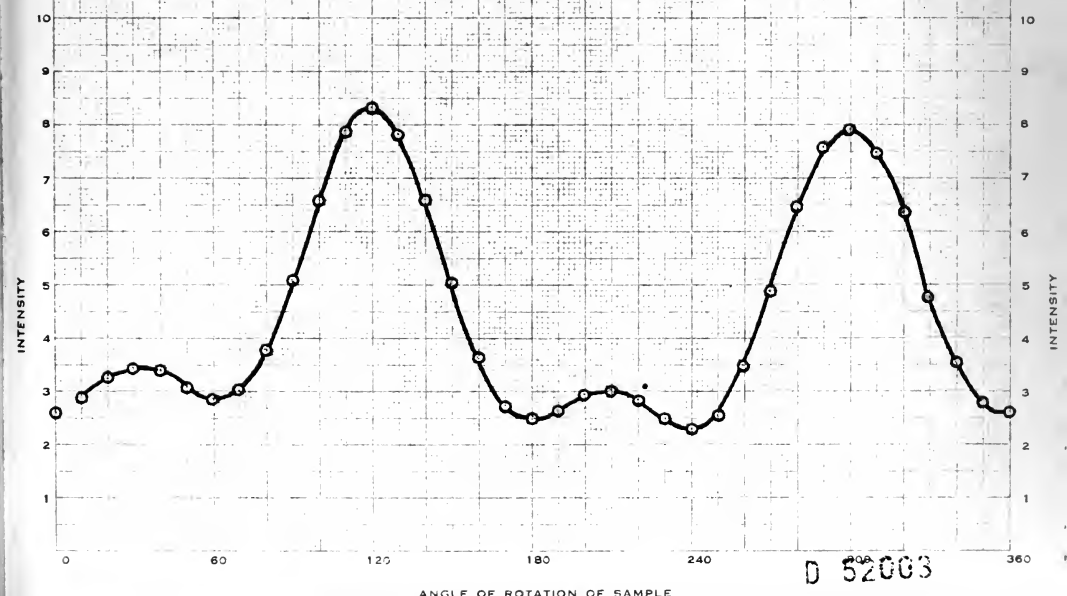
M_3	44.85	R_3	45.80
M_4	39.40	R_4	45.50
ΔM_{34}	5.45	ΔR_{34}	.20

Av ΔM 5.07 PHASE SHIFT

Av ΔR 0.17 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 1F 240 PPM N_2 DATE 5 APRIL 1952 AM

CRYSTAL No. 2 SURFACE FILM CONDITION 15th AUTOCLAVE OF 15 MIN. @ 233° C

	@ 355		@ 265		@ 175		@ 085	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	48.3	45.8	40.8	45.9	47.9	45.9	41.4	45.7
	48.1	45.9	41.0	45.9	47.8	45.9	41.5	45.8
Av.	48.20	45.85	40.90	45.90	47.85	45.90	41.45	45.75

M_1	48.20	R_1	45.85
M_2	40.90	R_2	45.90
ΔM_{12}	7.30	ΔR_{12}	-.05

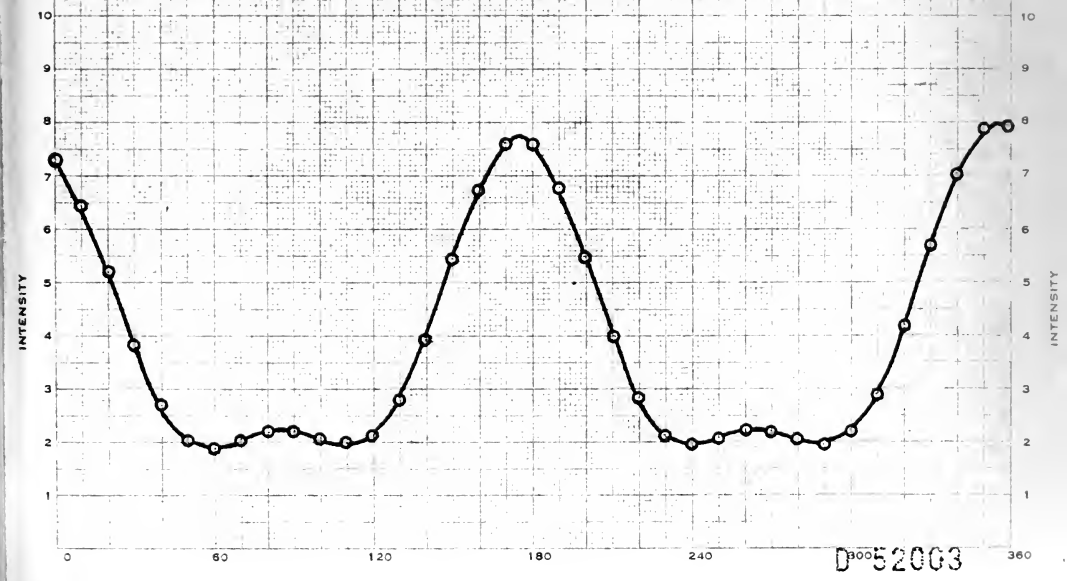
M_3	47.85	R_3	45.90
M_4	41.45	R_4	45.75
ΔM_{34}	6.40	ΔR_{34}	.15

Av ΔM 6.85 PHASE SHIFT

Av ΔR +0.05 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

Run #2

Corrosion film was grown at 295° C

D 52003

SAMPLE No 2A 6 PPM N_2 DATE 11 APRIL 1952 PM

CRYSTAL No 1 SURFACE FILM CONDITION 2nd ELECTROPOLISH

	@ 348		@ 078		@ 168		@ 258	
	M1	R1	M2	R2	M3	R3	M4	R4
	45.6	46.2	38.7	44.6	45.1	46.5	38.7	44.5
	45.5	46.3	39.0	44.5	45.0	46.4	38.7	44.5
Av.	45.55	46.25	38.85	44.55	45.05	46.45	38.70	44.50

M_1	45.55	R_1	46.25
M_2	38.85	R_2	44.55
ΔM_{12}	6.70	ΔR_{12}	1.70

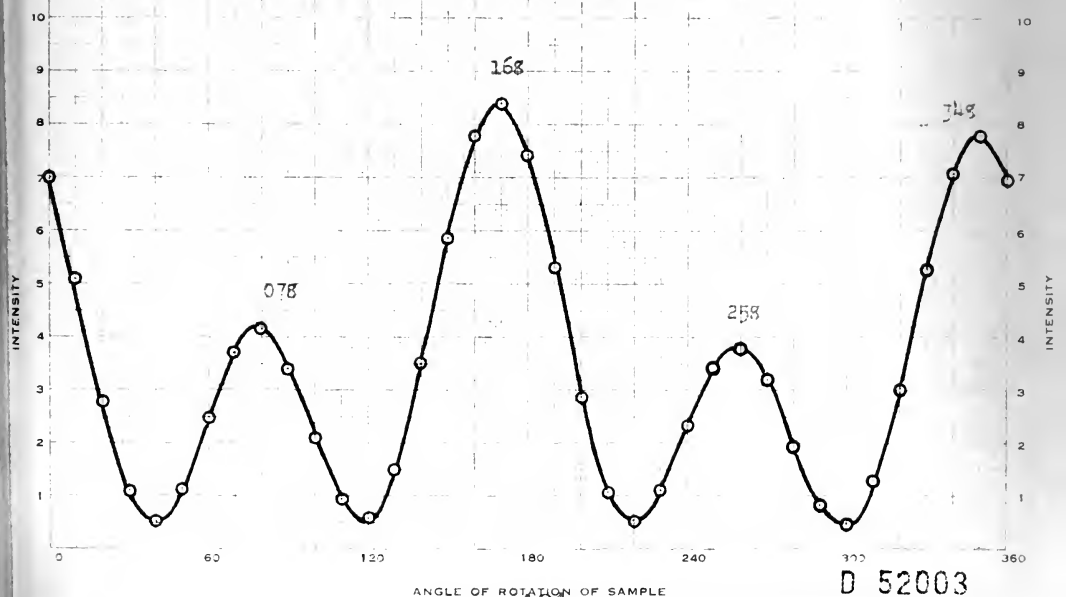
M_3	45.05	R_3	46.45
M_4	38.70	R_4	44.50
ΔM_{34}	6.35	ΔR_{34}	1.95

Av ΔM 6.53 PHASE SHIFT

Av ΔR 1.83 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 2A — 6 PPM N_2 — DATE 15 APRIL 1952 PM

CRYSTAL No. 1 — SURFACE FILM CONDITION 1st AUTOCLAVE OF 15 MIN. @ 295°C

	@ 064		@ 154		@ 244		@ 334	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	51.4	44.9	33.6	45.6	50.7	44.9	32.7	46.1
	51.3	44.9	33.6	45.7	50.7	44.7	32.7	46.0
Av.	51.35	44.90	33.60	45.65	50.70	44.80	32.70	46.05

M_1	51.35	R_1	44.90
M_2	33.60	R_2	45.65
ΔM_{12}	17.75	ΔR_{12}	- .75

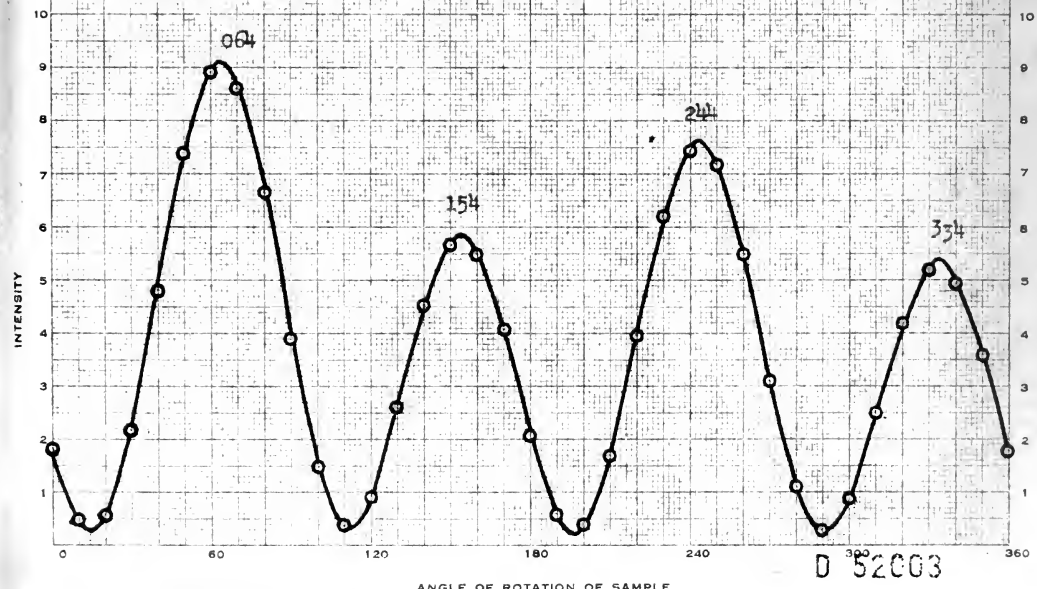
M_3	50.70	R_3	44.80
M_4	32.70	R_4	46.05
ΔM_{34}	18.00	ΔR_{34}	-1.25

Av ΔM 17.88 PHASE SHIFT

Av ΔR -1.00 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 2A 6 PPM N_2 DATE 15 APRIL AM 1952

CRYSTAL No. 2 SURFACE FILM CONDITION 2nd ELECTROPOLISH

	@ 326		@ 060		@ 148.5		@ 239	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	41.1	45.0	42.8	45.7	40.8	45.0	42.6	45.7
	41.1	45.0	42.8	45.7	41.0	45.0	42.4	45.6
Av.	41.10	45.00	42.80	45.70	40.90	45.00	42.50	45.65

M_1	41.10	R_1	45.00
M_2	42.80	R_2	45.70
ΔM_{12}	-1.70	ΔR_{12}	- .70

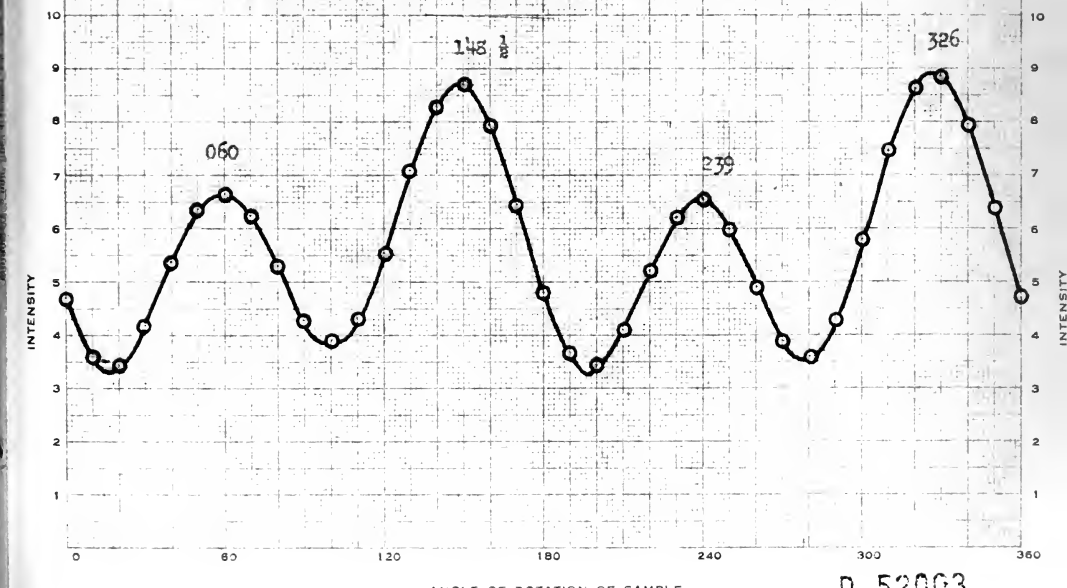
M_3	40.90	R_3	45.00
M_4	42.50	R_4	45.65
ΔM_{34}	-1.60	ΔR_{34}	- .65

Av ΔM -1.65 PHASE SHIFT

Av ΔR - .68 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 2A 6 PPM N_2 - DATE 15 APRIL 1952 PM

CRYSTAL No. 2 SURFACE FILM CONDITION 1st AUTOCLAVE OF 15 MIN. @ 295°C

	@ 321		@ 052		@ 140		@ 230	
	M1	R1	M2	R2	M3	R3	M4	R4
	44.6	50.1	42.3	40.9	44.2	50.1	42.1	40.6
	44.6	44.9	42.2	41.1	44.2	50.3	41.7	40.4
Av.	44.60	50.00	42.25	41.00	44.20	50.20	41.90	40.50

M_1	44.60	R_1	50.00
M_2	42.25	R_2	41.00
ΔM_{12}	2.35	ΔR_{12}	9.00

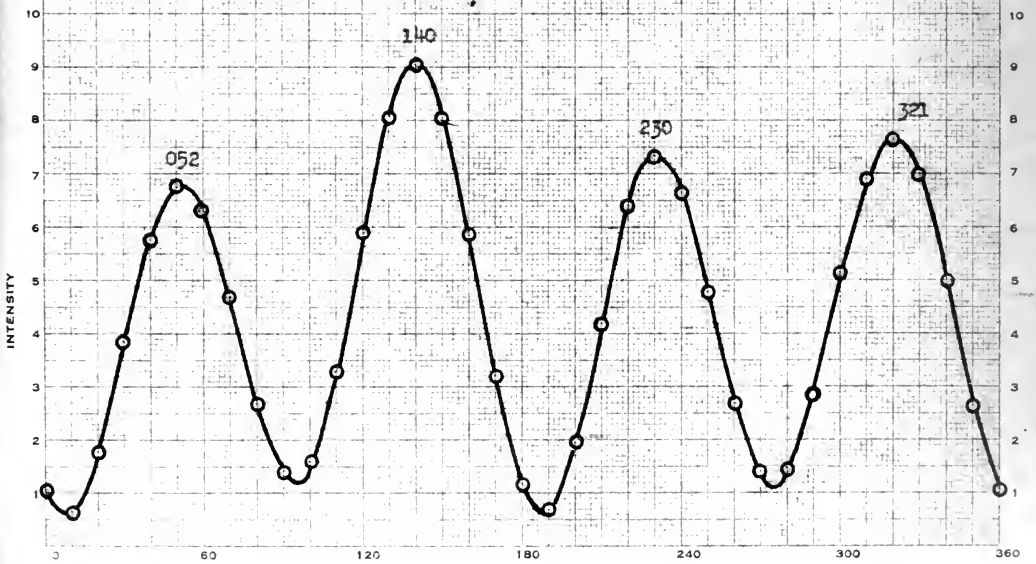
M_3	44.20	R_3	50.20
M_4	41.90	R_4	40.50
ΔM_{34}	2.30	ΔR_{34}	9.70

Av ΔM 2.33 PHASE SHIFT

Av ΔR 9.35 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No 3A2 6 RPM 72 DATE 17 APRIL 1952 AV

CRYSTAL No 2 SURFACE FILM CONDITION 3rd ELECTROLYTIC ETCH

	@ 333		@ 244		@ 150		@ 062	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	41.0	45.0	42.7	45.7	40.5	44.8	42.4	45.6
	41.0	45.0	42.5	45.7	40.7	45.0	42.3	45.6
Av.	41.05	45.00	42.60	45.70	40.60	44.90	42.35	45.60

M 41.05	R_1 45.00
M_2 42.60	R_2 45.70
ΔM_{12} -1.55	ΔR_{12} - .70

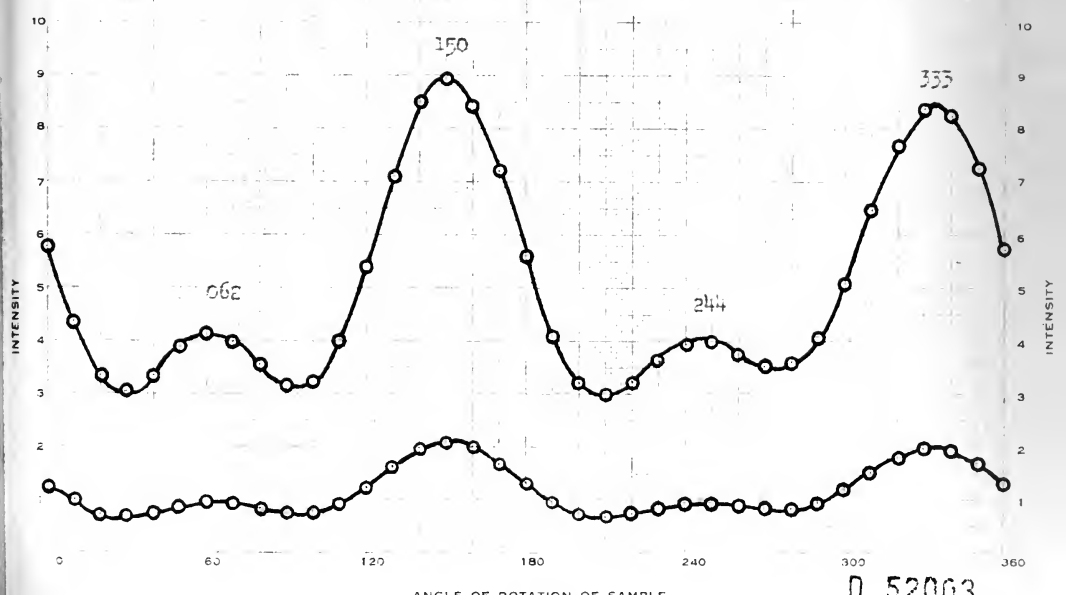
M_3 40.60	R_3 44.90
M_4 42.35	R_4 45.60
ΔM_{34} -1.75	ΔR_{31} - .70

Av ΔM -1.65 PHASE SHIFT

Av ΔR - .70 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 23 70 PPM N_2 DATE 11 MARCH 1958 AM

CRYSTAL No. 3 SURFACE FILM CONDITION [illegible]

	@ 307		@ 217		@ 127		@ 037	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	46.0	46.7	39.6	45.1	46.0	46.7	39.6	45.0
	46.0	46.7	39.4	45.1	46.0	45.7	39.6	45.0
Av.	46.00	46.70	39.50	45.10	46.00	46.70	39.60	45.00

M_1 46.00	R_1 46.70
M_2 39.50	R_2 45.10
ΔM_{12} 6.50	ΔR_{12} 1.60

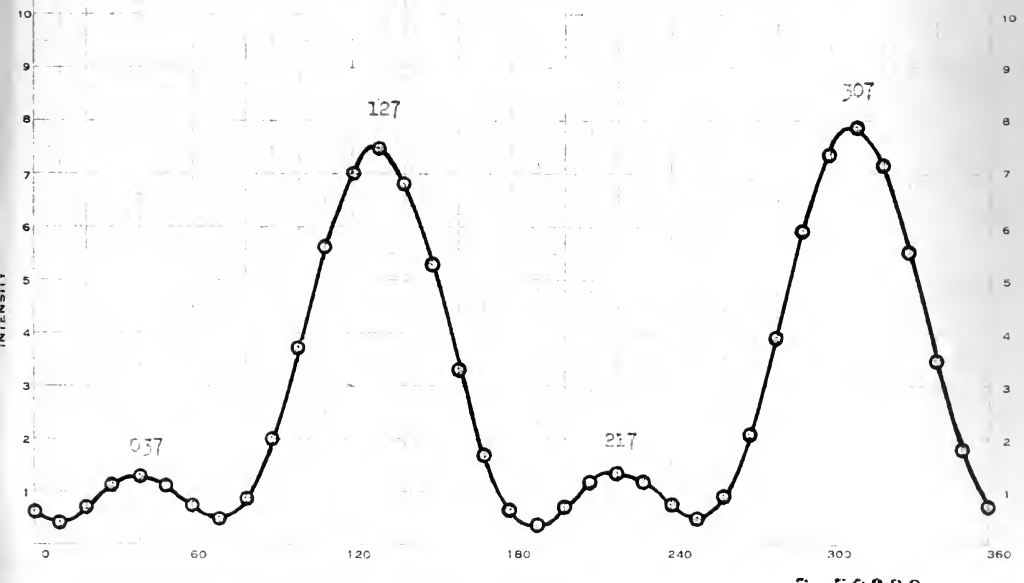
M_3 46.00	R_3 46.70
M_4 39.60	R_4 45.00
ΔM_{34} 6.40	ΔR_{34} 1.70

Av ΔM 6.45 PHASE SHIFT

Av ΔR 1.65 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 23 70 PPM H_2 DATE 15 APRIL 1952 PM

CRYSTAL No. 3 SURFACE FILM CONDITION 1st AUTOCLAVE 15 MIN. @ 295°C

	@ 243		@ 153		@ 063		@ 333	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	51.4	45.5	37.0	46.4	52.0	45.1	36.8	46.6
	51.8	45.4	36.5	46.4	51.8	44.9	37.0	46.4
Av.	51.60	45.45	36.75	46.40	51.90	45.00	36.90	46.50

M_1 51.60 R_1 45.45
M_2 36.75 R_2 46.40
ΔM_{12} 14.85 ΔR_{12} -0.95

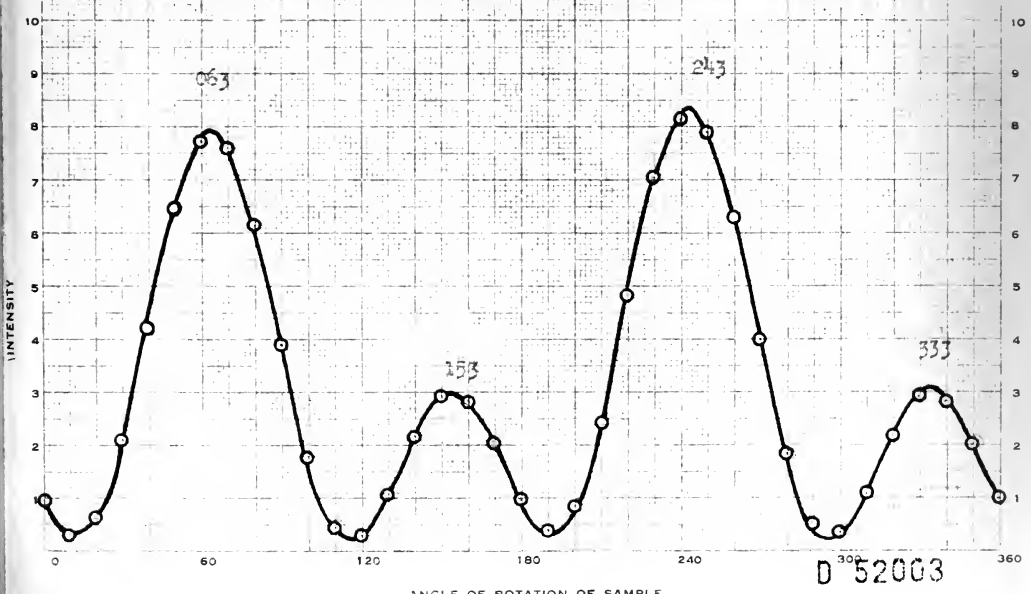
M_3 51.90 R_3 45.00
M_4 36.90 R_4 46.50
ΔM_{34} 15.00 ΔR_{34} -1.50

Av ΔM 14.92 PHASE SHIFT

Av ΔR -1.22 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 20 113 PPM N_2 DATE 12 APRIL 1952 AM

CRYSTAL No. 3 SURFACE FILM CONDITION 2nd ELECTROPOLISH

	@ 270		@ 001		@ 090		@ 180	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	45.3	46.6	40.8	44.7	45.1	46.6	40.5	45.0
	45.5	46.5	40.7	44.9	45.1	46.5	40.2	45.1
Av.	45.40	46.55	40.75	44.80	45.10	46.55	40.35	45.03

M_1	45.40	R_1	46.55
M_2	40.75	R_2	44.80
ΔM_{12}	4.65	ΔR_{12}	1.75

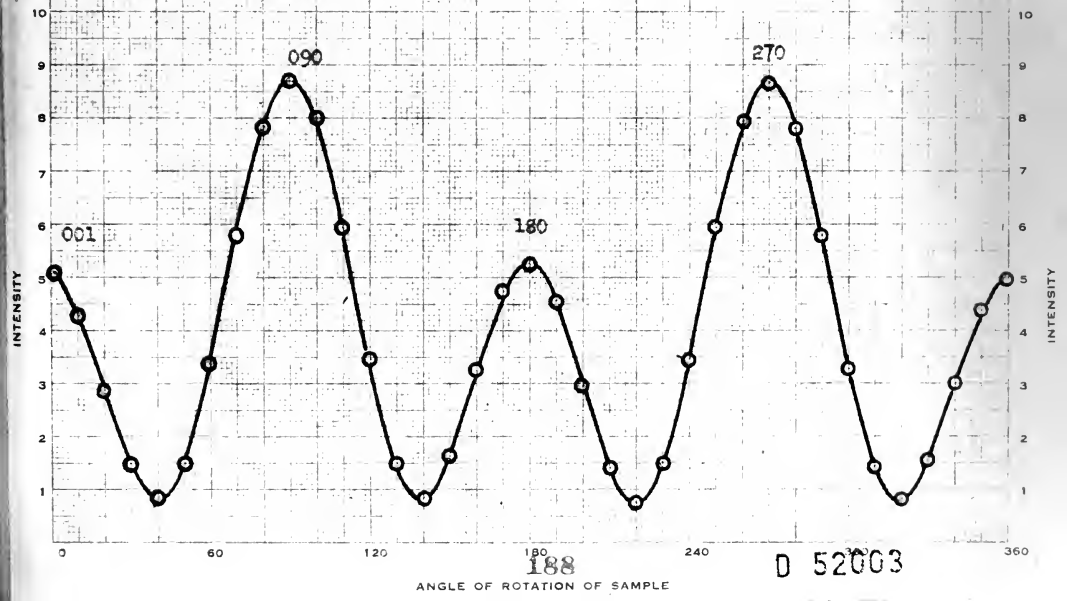
M_3	45.10	R_3	46.55
M_4	40.35	R_4	45.05
ΔM_{34}	4.75	ΔR_{34}	1.50

Av ΔM 4.70 PHASE SHIFT

Av ΔR 1.62 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 20 113 PPM N_2 DATE 16 APRIL 1952 PM

CRYSTAL No. 3 SURFACE FILM CONDITION 1st AUTOCLAVE OF 15 MIN. @ 295°C

	@ 009		@ 098		@ 188 ½		@ 278 ½	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	34.4	46.9	50.3	44.6	33.8	46.9	50.5	44.5
	34.0	47.0	50.0	44.6	33.7	46.8	50.4	44.6
Av.	34.20	46.95	50.15	44.60	33.75	46.85	50.45	44.55

M_1 34.20 R_1 46.95

M_2 50.15 R_2 44.60

ΔM_{12} -15.95 ΔR_{12} 2.35

M_3 33.75 R_3 46.85

M_4 50.45 R_4 44.55

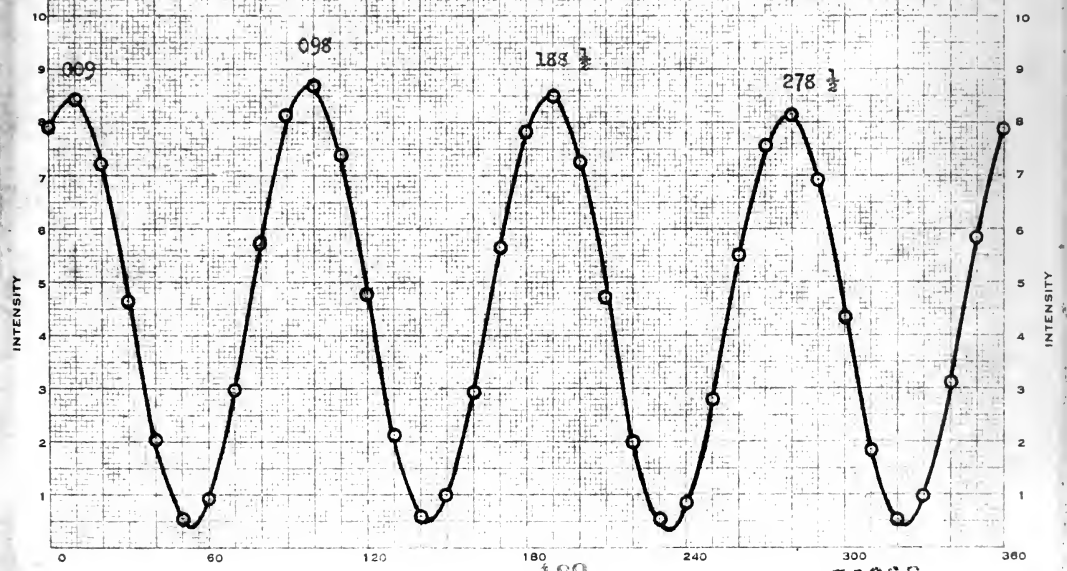
ΔM_{34} -16.70 ΔR_{34} 2.30

Av ΔM -16.33 PHASE SHIFT

Av ΔR 2.33 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 2D 147 PPM N_2 DATE 10 APRIL 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 2nd ELECTROPOLISH

	@ 337		@ 247		@ 157		@ 067	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	45.8	46.4	39.2	44.6	45.9	46.5	39.0	44.6
	45.8	46.5	39.3	44.6	45.9	46.5	39.0	44.6
Av.	45.80	46.45	39.25	44.60	45.90	46.50	39.00	44.60

M_1	45.80	R_1	46.45
M_2	39.25	R_2	44.60
ΔM_{12}	6.55	ΔR_{12}	1.85

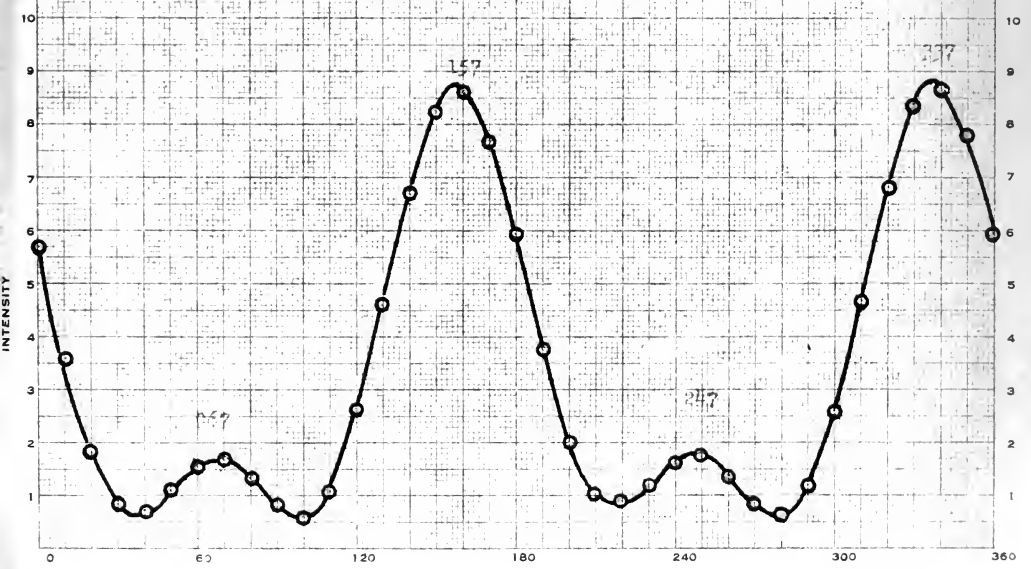
M_3	45.90	R_3	46.50
M_4	39.00	R_4	44.60
ΔM_{34}	6.90	ΔR_{34}	1.90

Av ΔM 6.72 PHASE SHIFT

Av ΔR 1.87 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 2D 147 PPM $\overline{X}_2$ DATE 15 April 1952 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 1st AUTOCLAVE 15 MIN. @ 295°C

@ 207		@ 117		@ 027		@ 297	
M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
52.3	45.3	36.4	46.1	52.3	45.4	35.8	46.0
52.1	45.3	36.3	46.1	52.1	45.2	36.1	46.0
Av. 52.20	45.30	36.35	46.10	52.20	45.30	35.95	46.00

M_1	52.20	R_1	45.30
M_2	36.35	R_2	46.10
ΔM_{12}	15.85	ΔR_{12}	= .80

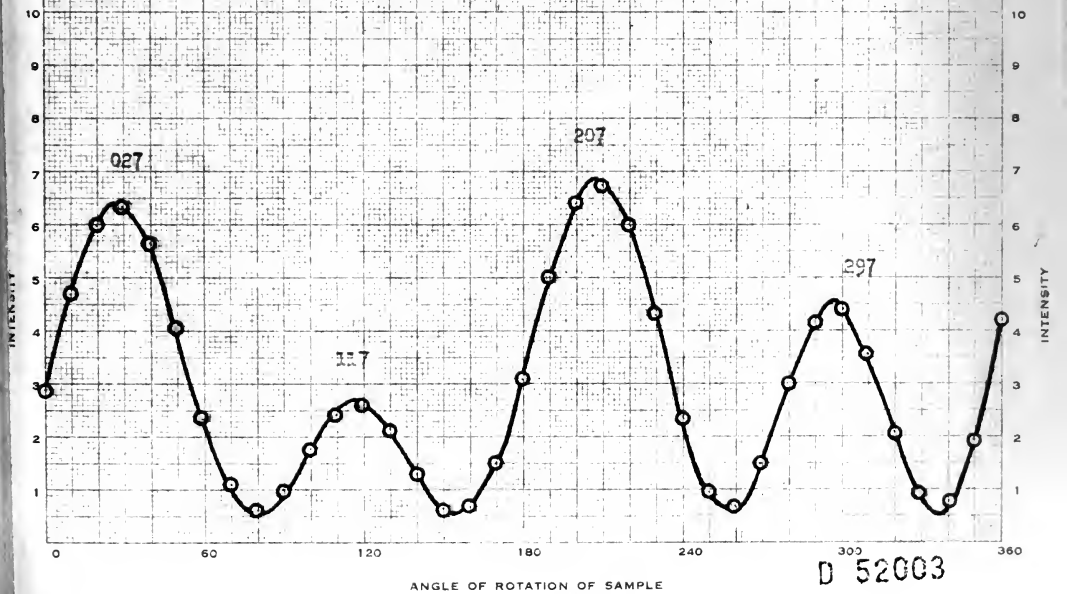
M_3	52.20	R_3	45.30
M_4	35.95	R_4	46.00
ΔM_{34}	16.25	ΔR_{34}	= .70

Av ΔM 16.05 PHASE SHIFT

Av ΔR -0.75 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No. 23 150 PPM N_2 DATE 15 APRIL PM

CRYSTAL No. 3 SURFACE FILM CONDITION 2nd ELECTROPOLISH

	@ 294		@ 024		@ 114		@ 204	
	M1	R1	M2	R2	M3	R3	M4	R4
	39.1	44.6	45.8	46.4	39.0	44.5	45.5	46.1
	39.0	44.5	45.7	46.2	39.1	44.6	45.5	46.4
Av.	39.05	44.55	45.75	46.30	39.05	44.55	45.50	46.25

M_1 39.05	R_1 44.55
M_2 45.75	R_2 46.30
ΔM_{12} -6.70	ΔR_{12} -1.85

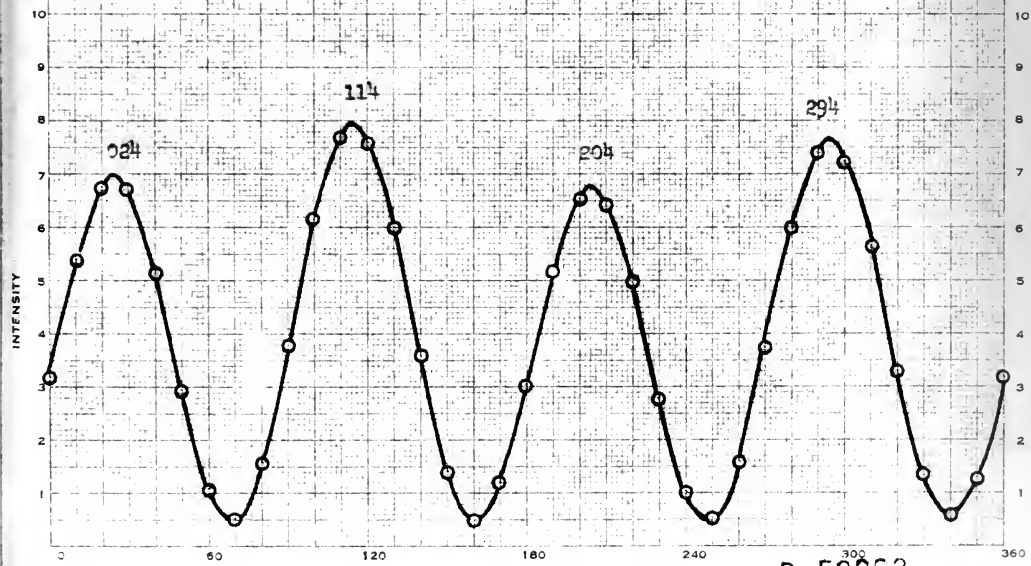
M_3 39.05	R_3 44.55
M_4 45.50	R_4 46.25
ΔM_{34} -6.45	ΔR_{34} -1.70

Av ΔM -6.58 PHASE SHIFT

Av ΔR -1.77 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

SAMPLE No [illegible] PPM [illegible] DATE [illegible]

CRYSTAL No. 3 SURFACE FILM CONDITION [illegible]

	@ 044		@ 133		@ 223		@ 313	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	32.4	40.8	51.6	39.3	32.0	41.6	51.8	39.2
	32.0	41.0	51.4	39.6	32.0	41.2	51.6	39.0
Av	32.20	40.90	51.50	32.45	32.00	41.40	51.70	39.10

M_1	32.20	R	40.90
M_2	51.50	R_2	39.45
ΔM_{12}	-19.30	ΔR	1.45

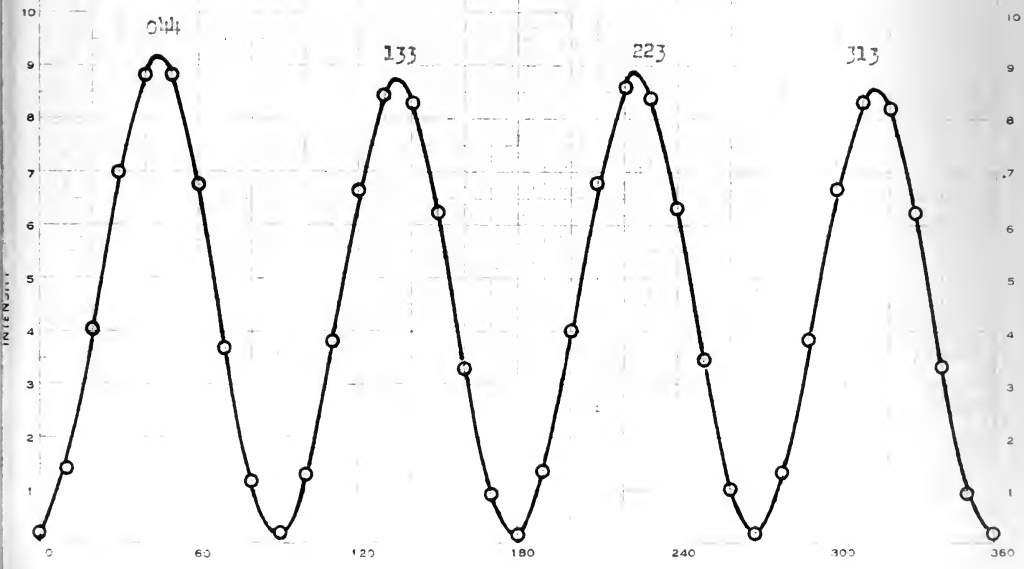
M_3	32.00	R_3	41.40
M_4	51.70	R_4	39.10
ΔM_{34}	-19.70	ΔR_{34}	2.30

Av ΔM -19.50 PHASE SHIFT

Av ΔR 1.87 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

D 52003

SAMPLE No. 2F 240 PPM N_2 DATE 15 APRIL 1952 AM

CRYSTAL No. 3 SURFACE FILM CONDITION 1st AUTOCLAVE 300°C at 15 MIN.

	@ 348		@ 258		@ 168		@ 078	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	50.7	45.7	36.9	45.5	49.1	45.2	36.2	45.4
	50.4	45.5	37.0	45.2	49.2	45.1	36.1	45.5
	50.6	45.3						
Av.	50.57	45.50	36.95	45.35	49.15	45.15	36.15	45.45

M_1	50.57	R_1	45.50
M_2	36.95	R_2	45.35
ΔM_{12}	13.62	ΔR_{12}	.15

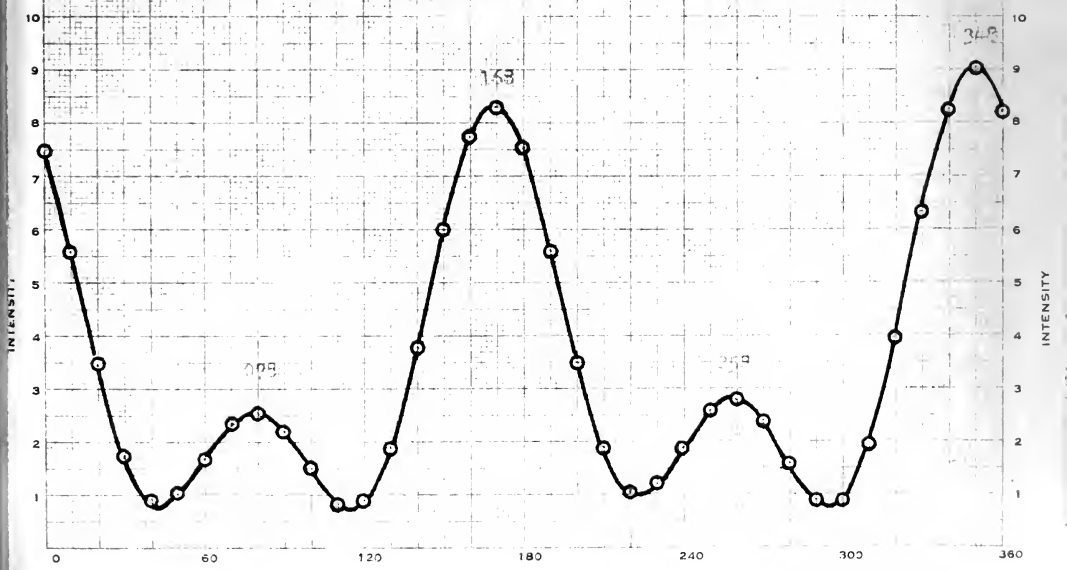
M_3	49.15	R_3	45.15
M_4	36.15	R_4	45.45
ΔM_{34}	13.00	ΔR_{34}	-.30

Av ΔM 13.31 PHASE SHIFT

Av ΔR -0.08 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



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SAMPLE No. 2F 240 PPM N_2 DATE 11 APRIL 1952 PM

CRYSTAL No. 3 SURFACE FILM CONDITION 2nd ELECTROPOLISH

	@ 313		@ 223		@ 133		@ 043	
	M1	R1	M2	R2	M3	R3	M4	R4
	46.4	46.5	39.7	44.7	46.5	46.6	39.5	44.8
	46.6	46.6	39.6	44.7	46.3	46.6	39.3	44.8
Av.	46.50	46.55	39.65	44.70	46.40	46.60	39.40	44.80

M_1	46.50	R_1	46.55
M_2	39.65	R_2	44.70
ΔM_{12}	6.85	ΔR_{12}	1.85

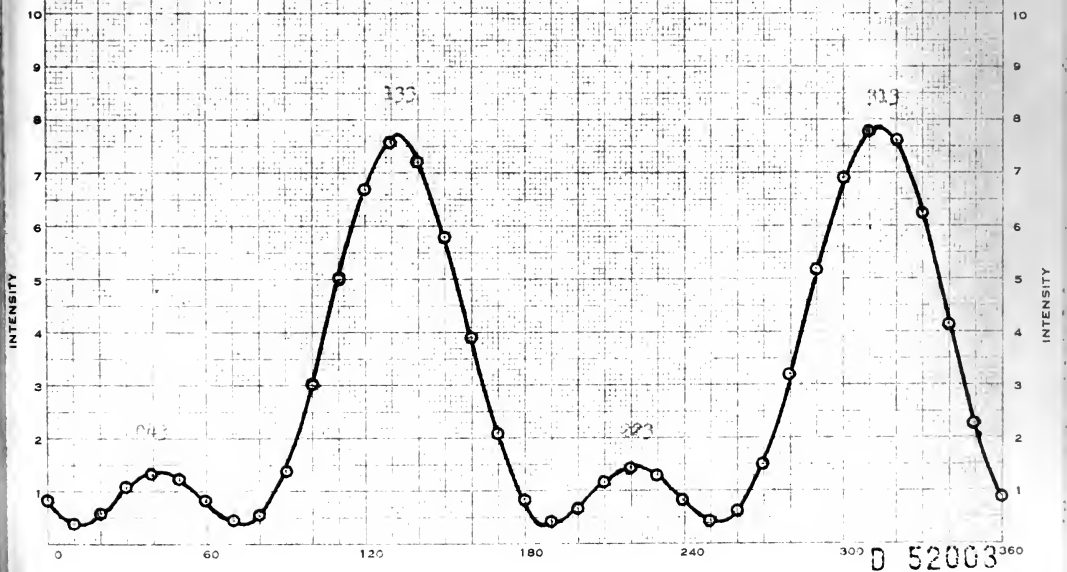
M_3	46.40	R_3	46.60
M_4	39.40	R_4	44.80
ΔM_{34}	7.00	ΔR_{34}	1.80

Av ΔM 6.92 PHASE SHIFT

Av ΔR 1.82 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



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SAMPLE No. 2F 240 PPM N_2 DATE 11 APRIL 1952 PM

CRYSTAL No. 4 SURFACE FILM CONDITION 2nd ELECTROPOLISH

	@ 286		@ 196		@ 106		@ 016	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	43.5	45.8	41.3	45.0	43.6	45.8	41.2	45.1
	43.5	45.9	41.3	45.0	43.5	45.9	41.1	45.1
Av.	43.55	45.85	41.30	45.00	43.55	45.85	41.15	45.10

M_1	43.55	R_1	45.85
M_2	41.30	R_2	45.00
ΔM_{12}	2.25	ΔR_{12}	.85

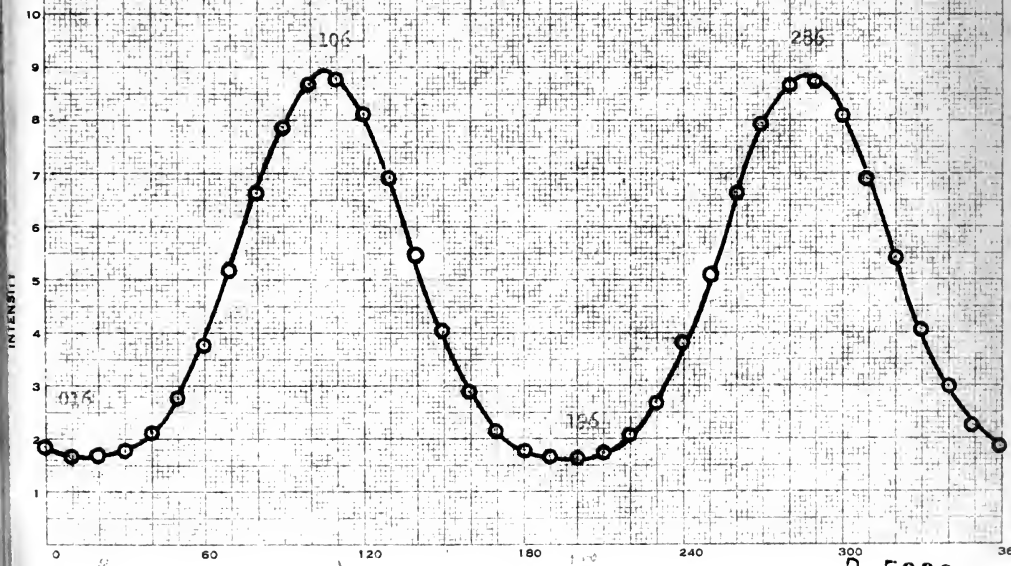
M_3	43.55	R_3	45.85
M_4	41.15	R_4	45.10
ΔM_{34}	2.40	ΔR_{34}	.75

Av ΔM 2.32 PHASE SHIFT

Av ΔR 0.80 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



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SAMPLE No. 2F 240 PPM N_2 DATE 15 APRIL 1952 PM

CRYSTAL No. 4 SURFACE FILM CONDITION 1st AUTOCLAVE 15 MIN. @ 295°C

	@ 324		@ 234		@ 144		@ 054	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	43.7	48.2	41.6	43.3	44.7	48.5	41.8	42.9
	43.9	48.2	41.5	43.4	44.5	48.6	41.6	43.1
Av.	43.80	48.20	41.55	43.35	44.60	48.55	41.70	43.00

M_1	43.80	R_1	48.20
M_2	41.55	R_2	43.34
ΔM_{12}	2.25	ΔR_{12}	4.85

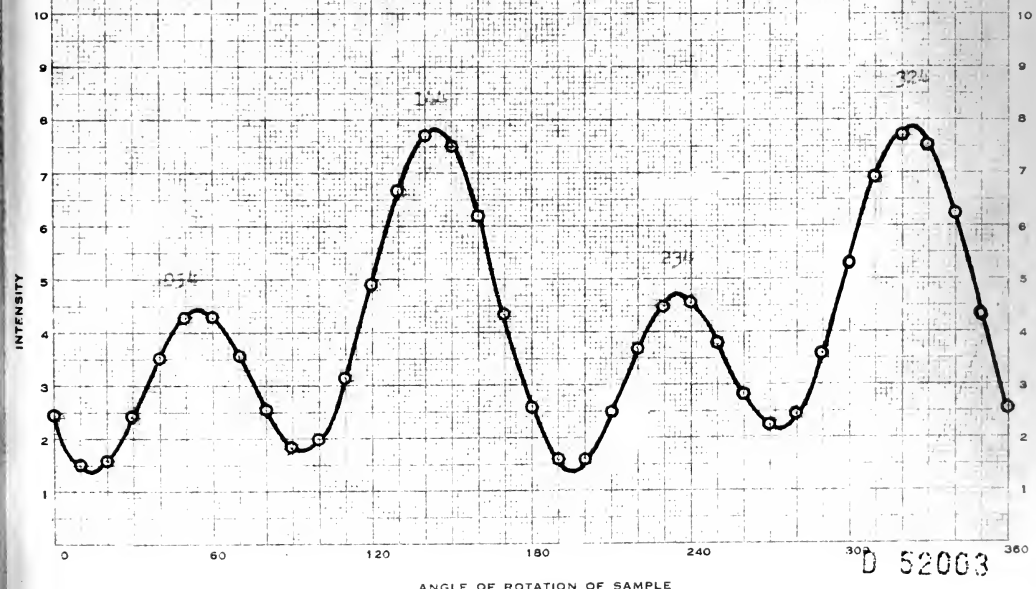
M_3	44.60	R_3	48.55
M_4	41.70	R_4	43.00
ΔM_{34}	2.90	ΔR_{34}	5.55

Av ΔM 2.58 PHASE SHIFT

Av ΔR 5.20 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



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SAMPLE No. 3F 240 PPM N_2 DATE 17 APRIL 1952 PM

CRYSTAL No. 4 SURFACE FILM CONDITION 3rd ELECTROPOLISH

	@ 314		@ 224		@ 134		@ 044	
	M_1	R_1	M_2	R_2	M_3	R_3	M_4	R_4
	41.8	45.3	43.6	46.1	41.4	45.3	43.5	46.0
	41.6	45.3	43.6	46.0	41.6	45.3	43.4	46.0
Av.	41.70	45.30	43.60	46.05	41.50	45.30	43.45	46.00

M_1	41.70	R_1	45.30
M_2	43.60	R_2	46.05
ΔM_{12}	-1.90	ΔR_{12}	- .75

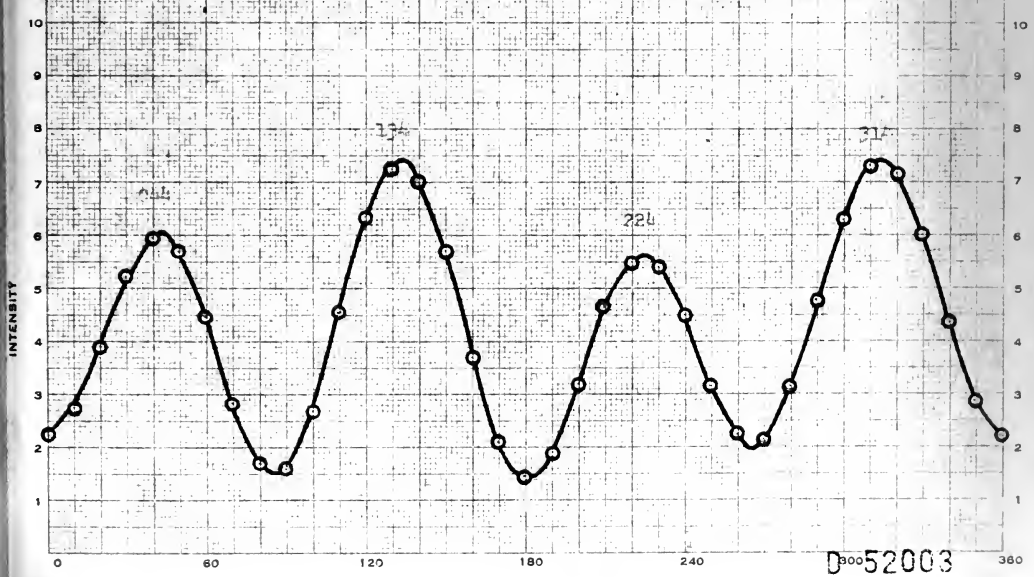
M_3	41.50	R_3	45.30
M_4	43.45	R_4	46.00
ΔM_{34}	-1.95	ΔR_{34}	- .70

Av ΔM -1.92 PHASE SHIFT

Av ΔR -0.72 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



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Appendix E

Summary of Data

The data are summarized in four sets of curves as follows:

1. Run #1
   (a) Faired plot of phase shift versus number of autoclavings.
   (b) Faired plot of rotation of plane of polarization versus number of autoclavings.

2. Run #2
   (a) Faired plot of phase shift versus number of autoclavings.
   (b) Faired plot of rotation of plane of polarization versus number of autoclavings.

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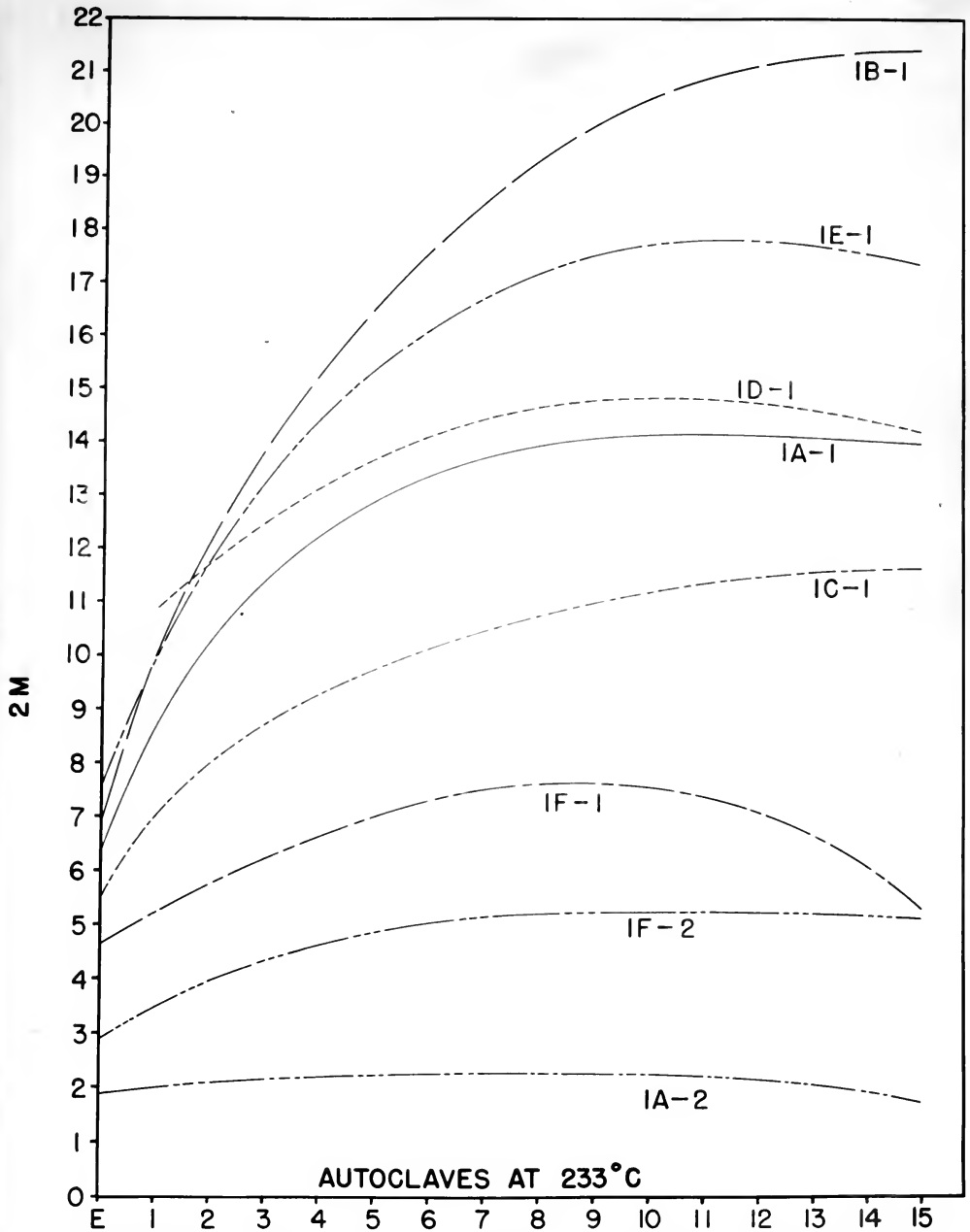
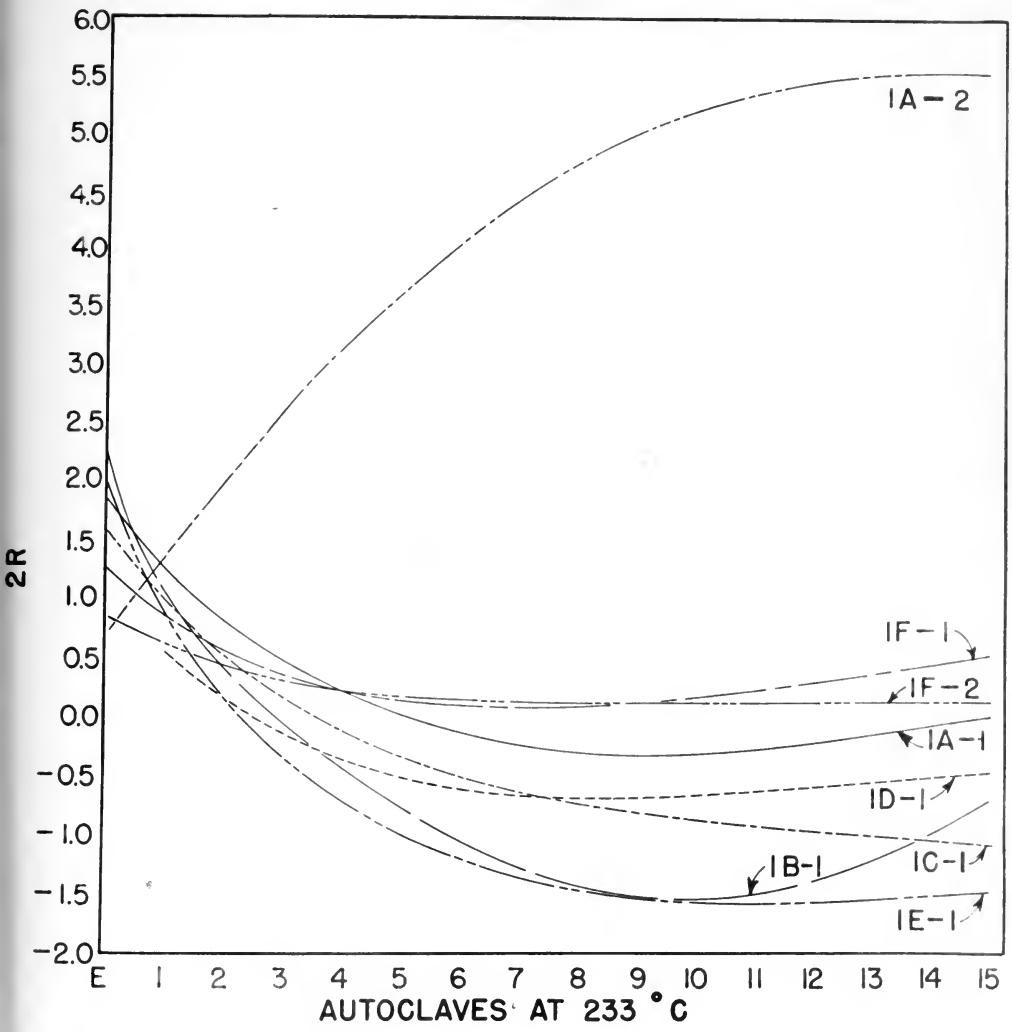


SUMMARY PLOT OF PHASE SHIFTS VERSUS CORROSION TIME

MET LAB
JWH NRN 23APR52

160J-1



SUMMARY PLOT OF ROTATION OF PLANE OF POLARIZATION VS. CORROSION TIME

T LAB
H NRN 23 APR 52

16032 − 1

Appendix F

Calculation of Characteristic Angle

$$\tan \tau = -0.8666 \frac{\sin 2m}{\sin 2r} + 0.0208$$

where τ is the characteristic angle

2m is the phase shift in the reflected light

2r is the rotation in the reflected light

and the constants are those given by Bausch and Lomb for Compensator No. 37 with Zirconarc white light.

Five place values for sin 2m and sin 2r were used in calculations. 2m, 2r, and τ are tabulated in Appendix F for each crystal in the electropolished condition and after each autoclaving. Plots of the characteristic angle versus corrosion time are contained in Chapter IV.

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RUN #1

	141			142		
	2m	2r	Σ	2n	2r	Σ
E	6.30	1.87	294-14	1.88	0.70	299-28
1	8.70	1.25	282-15	2.02	1.36	316-00
2	10.30	0.80	276-42	2.12	2.00	325-52
3	11.43	0.48	273-38	2.18	2.61	331-45
4	12.27	0.22	271-33	2.23	3.15	335-43
5	12.90	0.02	270-08	2.27	3.62	338-20
6	13.37	-0.12	269-13	2.30	4.05	337-45
7	13.70	-0.23	268-35	2.31	4.45	341-56
8	13.91	-0.30	268-08	2.32	4.77	343-05
9	14.03	-0.32	268-01	2.32	5.02	343-56
10	14.09	-0.30	268-09	2.30	5.21	342-31
11	14.10	-0.27	268-20	2.26	5.35	345-22
12	14.09	-0.21	268-42	2.18	5.45	346-11
13	14.05	-0.15	269-04	2.08	5.51	347-00
14						
15	13.95	0.00	270-00	1.80	5.55	348-48

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D 5200

RUN #1

	1B1			1C1		
	2m	2r	τ	2m	2r	τ
E	6.77	2.03	294-27	5.38	1.80	295-52
1	10.00	1.05	279-01	7.03	0.97	281-45
2	12.16	0.42	272-59	8.65	0.53	275-41
3	13.85	-0.03	268-08	8.85	0.17	271-40
4				9.35	-0.12	268-53
5	16.47	-0.77	265-56	9.73	-0.33	267-05
6	17.58	-1.07	264-45	10.14	-0.51	265-40
7	18.52	-1.28	264-00	10.47	-0.64	264-45
8	19.28	-1.42	263-36	10.76	-0.74	264-05
9	19.93	-1.55	263-15	11.00	-0.82	263-36
10	20.45	-1.55	263-24	11.18	-0.88	263-15
11	20.62	-1.51	263-37	11.33	-0.93	262-57
12	21.10	-1.40	264-12	11.45	-0.98	262-39
13	21.27	-1.23	264-57	11.55	-1.01	262-30
14						
15	21.45	-0.72	267-05	11.65	-1.08	262-03

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RUN #1

	1D1			1W1		
	2m	2r	℧	2m	2r	℧
E	9.90	1.10	291-24	7.55	2.00	291°-49'
1	10.87	0.55	274-22	9.35	.82	277°-58'
2	11.73	0.15	271-06	11.80	.20	271°-28'
3	12.50	-0.15	268-58	13.25	-.35	268°-28'
4	13.13	-0.36	267-38	14.45	-.70	265°-48'
5	13.67	-0.51	266-46	15.35	-.98	264°-29'
6	14.10	-0.61	266-15	16.10	-1.18	263°-40'
7	14.40	-0.67	265-58	16.67	-1.34	263°-03'
8	14.62	-0.70	265-52	17.15	-1.44	262°-44'
9	14.75	-0.70	265-62	17.50	-1.52	262°-29'
10	14.79	-0.68	266-01	17.70	-1.57	262°-20'
11	14.77	-0.65	266-11	17.80	-1.58	262°-20'
12	14.70	-0.61	266-24	17.77	-1.57	262°-21'
13	14.58	-0.57	266-37	17.68	-1.56	262°-22'
14						
15	14.20	-0.48	267-04	17.35	-1.50	262°-31'

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RUN #1

	1F1			1F2		
	2m	2r	℄	2m	2r	℄
E	4.65	1.25	292°-08'	2.90	.85	295°-26'
1	5.24	.85	285°-45'			
2	5.77	.55	278°-10'	5.95	.45	279°-18'
3	6.24	.35	274°-49'	4.82	.30	275°-58'
4	6.65	.21	272°-45'	4.62	.21	273°-55'
5	7.00	.12	271°-29'	4.85	.17	273°-01'
6	7.27	.09	271°-04'	5.01	.15	272°-14'
7	7.48	.09	271°-02'	5.12	.12	272°-01'
8	7.62	.10	271°-08'	5.18	.12	272°-00'
9	7.65	.12	271°-21'	5.25	.11	271°-40'
10	7.60	.17	271°-56'	5.24	.11	271°-40'
11	7.35	.22	272°-35'	5.25	.12	271°-59'
12	7.05	.30	273°-40'	5.21	.12	271°-59'
13	6.67	.37	274°-46'	5.18	.12	272°-00'
14						
15	5.30	.51	278°-15	5.13	.13	272°-11'

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RUN #2

	2A1			2A2		
	2m	2r	℄	2m	2r	℄
E	6.55	1.83	293°-00'	-1.65	0.68	258°-36'
1	17.88	-1.00	265°-08'	2.33	9.35	351°-41'

	2E3			2C3		
E	6.45	1.65	291°-10'	4.70	1.62	297°-27'
1	14.92	-1.22	262°-57'	-13.83	2.35	257°-30'

	2D1			2D3		
E	6.72	1.87	291°-24'	-6.58	-1.77	292°-10'
1	16.05	-0.75	265°-58'	-19.50	1.87	261°-41'

	2F3			2F4		
E	6.92	1.82	291°-43'	2.32	0.80	297°-36'
1	15.31	-0.08	269°-29'	2.58	9.20	342°-45'

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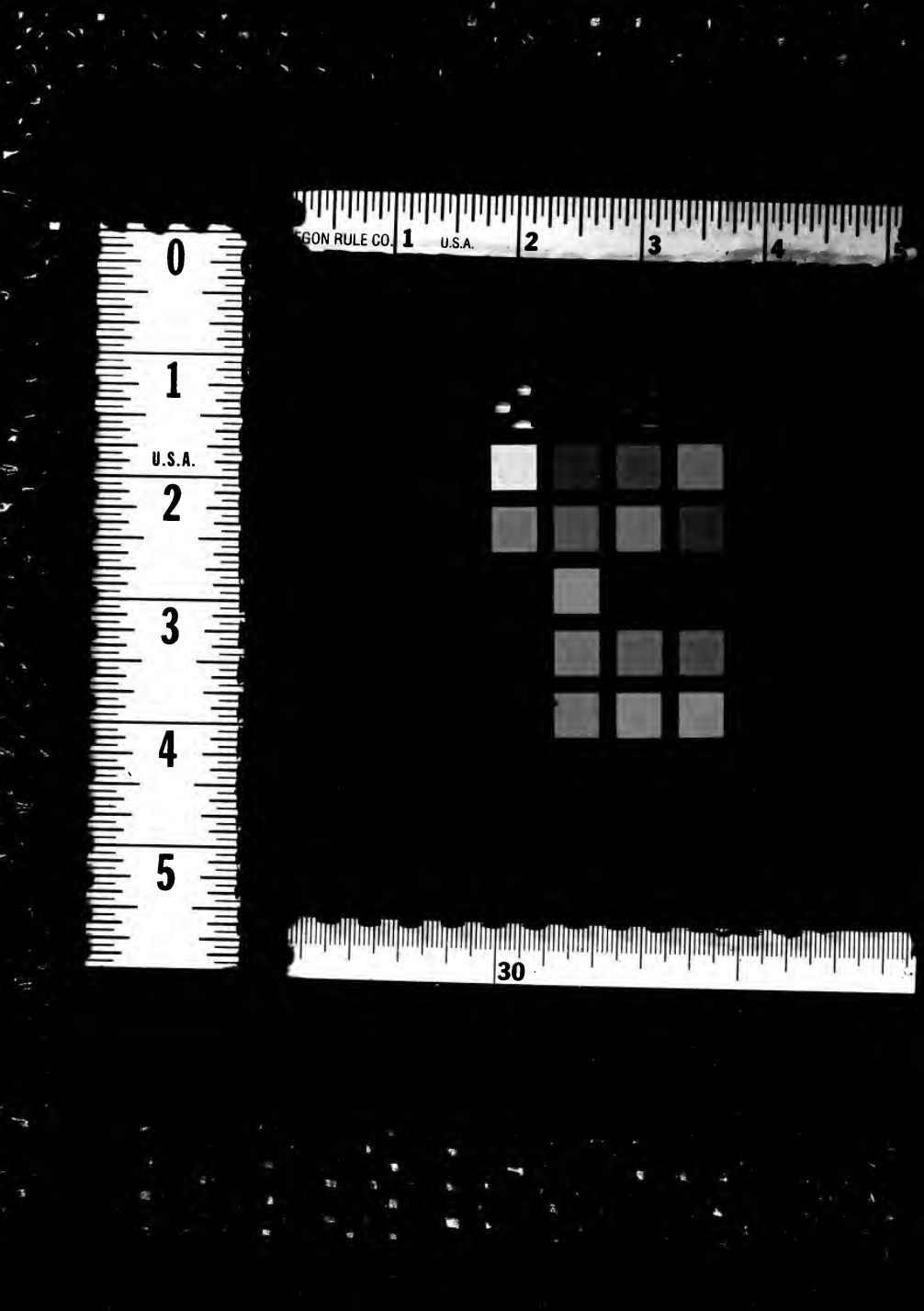
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